Title:
Selective Laser Melting Inconel 718 Restart Study

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Executive Summary: (Purpose and Result)

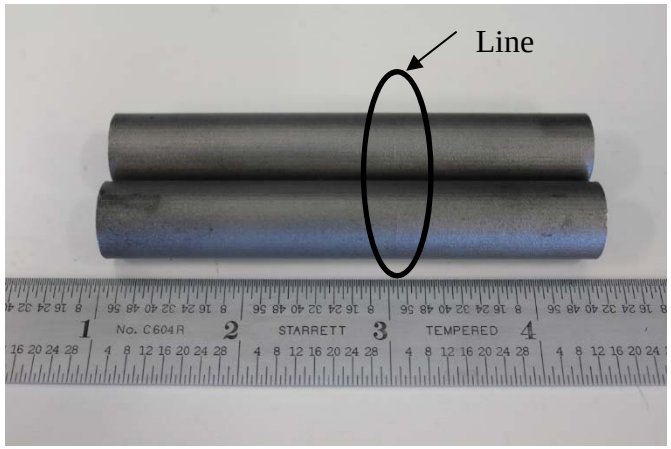
Selective Laser Melting (SLM) machines can occasionally shut down (such as in the event of a power failure), which requires the SLM build to be restarted. It has been observed that machine restarts can produce visible “restart lines” in the part, and, in at least one instance, a test specimen containing a restart line exhibited poor mechanical properties. The goal of this study is to evaluate the effect of different machine restart factors on the mechanical properties of the produced SLM material. Test specimens were produced by varying time to restart, the number of re-sinters, and whether or not the build required re-levelling. These are build variables that are typically controlled by the machine operator. Test results indicate that re-leveling and using three re-sinters produces acceptable material. Statistical analysis indicates that re-leveling and re-sintering have strong effects on the resulting material properties. Additionally, the visibility of a restart line loosely correlates with material properties such that obvious restart lines tended to indicate worse material properties.

References: (work orders, reports, etc.)

Additive Manufacturing Restart Conditions Investigation Test Plan by William G. Tilson (ESSSA-FY15-338), Test Report #2015-0286 Seq 3, Test Report #2015-0286 Seq 4, Test Report #2014-0537 Seq 4, Personal Conversation with Dennis Lambert (EM20) and Charles Martin (ER51).

Background

- During the course of a Selective Laser Melting (SLM) build, the machine has the potential to stop, requiring that the build process be restarted to continue production.
- In some cases, these machine restarts are identified in the final parts by “witness lines” at the point at which the machine stopped.
- In at least one instance, a witness line is believed to be the point of a premature tensile test specimen failure.
- However, the presence of a witness line does not necessarily indicate that the test specimen will produce poor tensile properties, opening the possibility that machine restarts can be done in such a manner as to have minimal effect on the produced material.
- A study was devised to investigate the effect of machine restarts on material properties.



Machine Restart Variables

1. **Time to restart** – the amount of time between a machine stoppage and a restart. The time to restart can vary from minutes to days.
2. **Number of re-sinters**– as part of the restart process, the layer of sintered metal that was deposited right before the machine stoppage is re-traced, or "re-sintered", by the laser. This occurs at least once, but can be made to occur several times.
3. **Re-leveling** – under some circumstances, the build stage will not maintain the same Z-axis position after a stoppage. If this occurs, the stage needs to be moved, or "re-leveled", to the Z-axis position of the previous layer before continuing the build. Not all restarts require re-leveling.
4. **Inert gas purge** – during the build process, an inert gas is circulated through the build chamber. After a machine stoppage, the purge gas flow typically stops.

- These variables are typically controllable by the machine operator.

Machine Restart Variables

1. **Surface area of restart plane –** the area of sintered metal that was deposited in the layer before a machine stoppage. This is dependent on the build part.
2. **Cooling rate –** the rate at which the part cools after a stoppage. Cooling rate is affected by many factors, including time, atmosphere, and the size of the build part.
3. **Oxidation –** depending on the conditions in the machine, oxidation can occur on the restart plane during a machine stoppage.

- These variables are typically uncontrollable; they are affected by the build part geometry and the machine.

Test Plan

- **Objective:** Determine the effect of controllable restart variables on tensile properties.

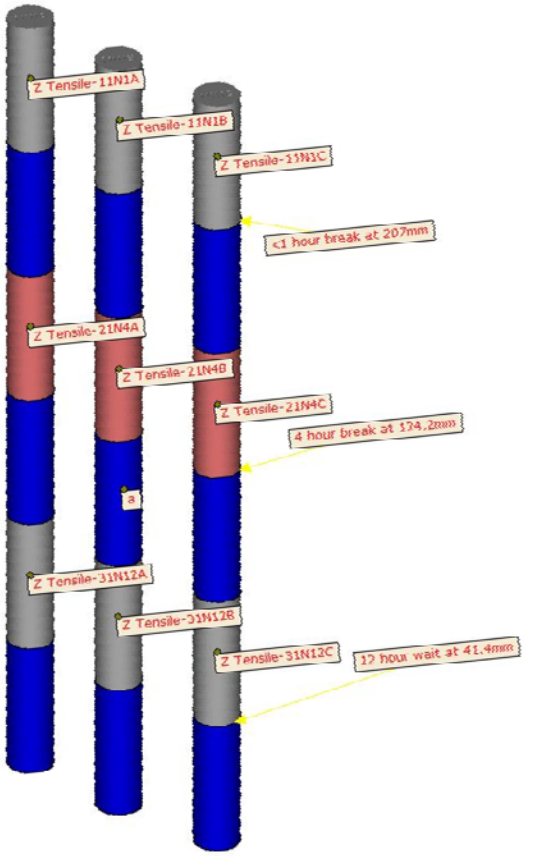
- Three parameters are varied to either two or three levels:
  - Time to restart: < 1 hour, 4 hours, or 12 hours
  - Re-leveling: Yes (required re-level) or No (did not require re-level)
  - Re-sinters: 1 or 3

- 12 total runs were produced with three test specimens per run.
- Test specimens were produced from Inconel 718 on the M1 SLM machine using standard parameters.
- The build direction was Z (vertical).
- The inert purge gas was off during the machine stoppage.
- All specimens received the standard SLM Inconel 718 heat treatment, including stress relief, hot isostatic press (HIP), and solution and precipitation heat treatments per AMS 5664E. See appendices for heat treatment details.
- Specimens were tensile tested in accordance with ASTM E8.

Run	Re-sinters	Re-level	Time to Restart (hours)
1	1	No	< 1
2	1	No	4
3	1	No	12
4	1	Yes	< 1
5	1	Yes	4
6	1	Yes	12
7	3	No	< 1
8	3	No	4
9	3	No	12
10	3	Yes	< 1
11	3	Yes	4
12	3	Yes	12

SLM Build

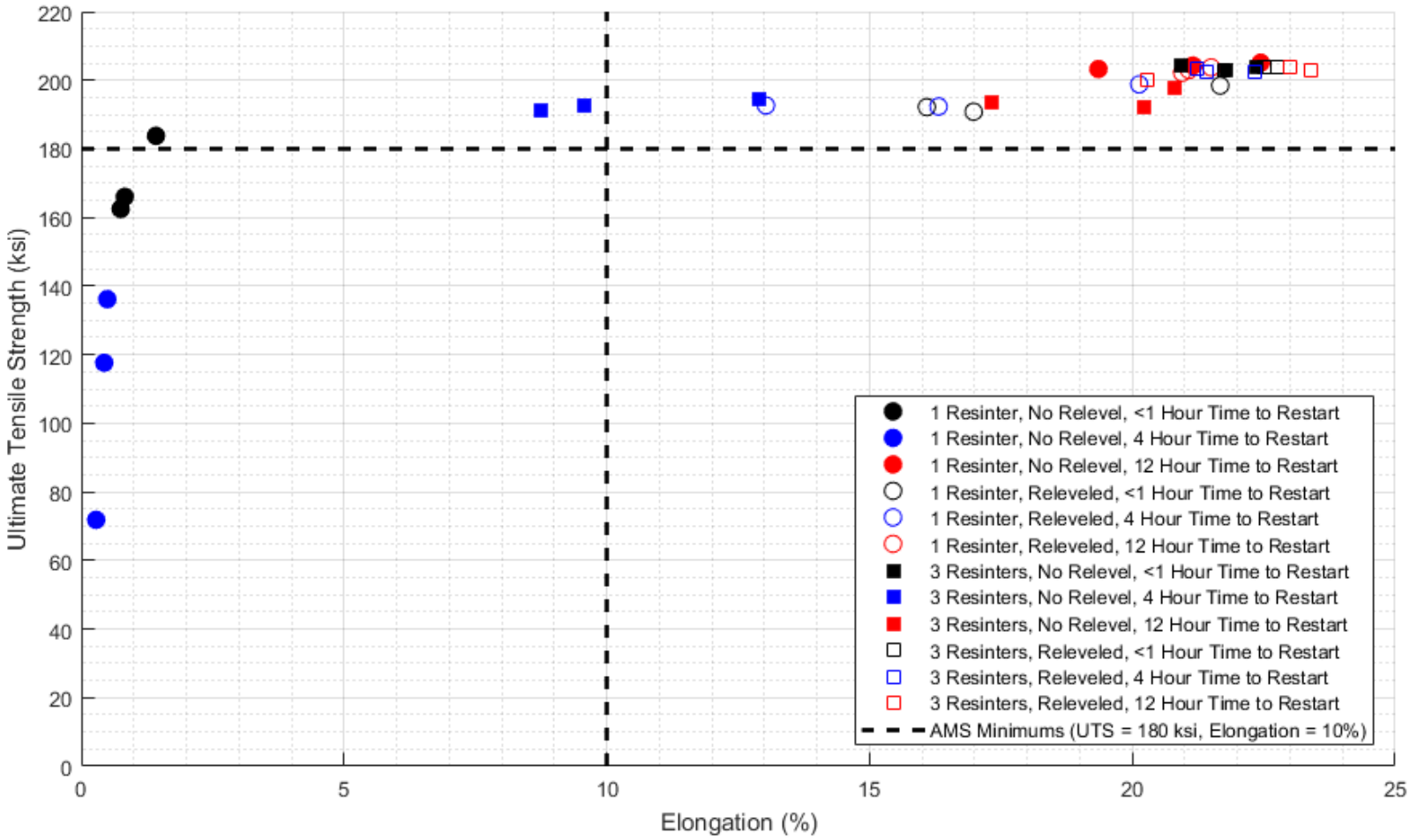
- The specimens were produced in five separate builds. An example build is shown here.
  - The fourth build crashed during the final <1 hour break (Run 10). The Run 10 specimens were completed on a fifth build.
- Re-level and re-sinter parameters were not varied within a build; only time to restart varied within builds.
- Four confidence tensile specimens were included in the fourth build to witness the heat treatment operations.



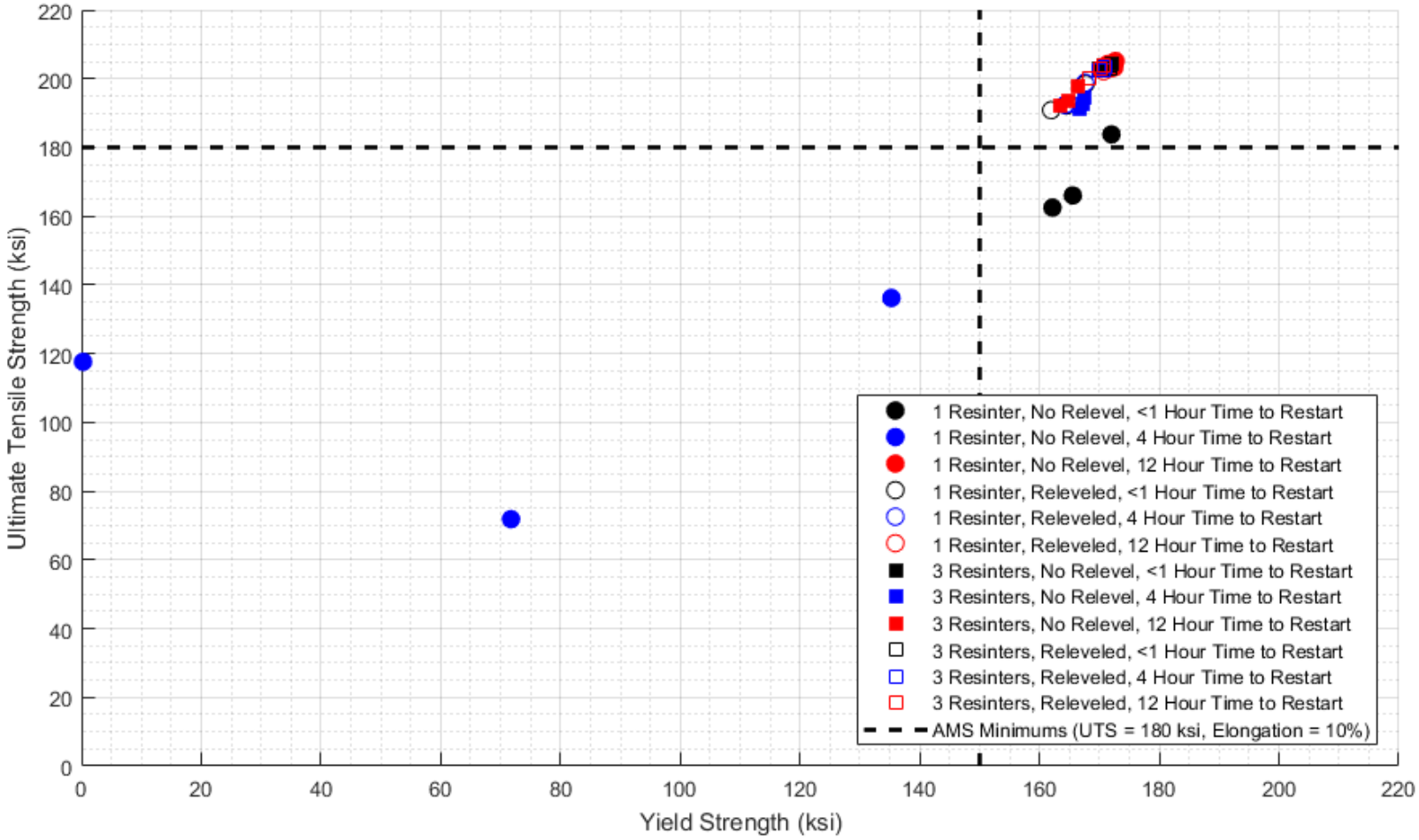
Results

- AMS 5664E establishes minimum requirements for ultimate tensile strength, yield strength, and total elongation. The next two pages show plots of tensile test results in terms of these minimum requirements.
- At least one specimen from three of the runs fall below the AMS standard minimum requirements for Inconel 718 tensile properties.
    - Run 1 falls below the ultimate strength and total elongation requirements, Run 2 falls below the minimum in all three properties, and Run 8 fails the total elongation requirement.
- Regardless, as the strength properties for most specimens are well above the minimum requirements, these results are indicative of a proper heat treatment.
- Previous testing has shown that build defects can be present even when properties are well above the minimum AMS requirements.

Results


Ultimate Tensile Strength (ksi)
Elongation (%)
0
20
40
60
80
100
120
140
160
180
200
220
0
5
10
15
20
25
1 Resinter, No Relevel, <1 Hour Time to Restart
1 Resinter, No Relevel, 4 Hour Time to Restart
1 Resinter, No Relevel, 12 Hour Time to Restart
1 Resinter, Releveled, <1 Hour Time to Restart
1 Resinter, Releveled, 4 Hour Time to Restart
1 Resinter, Releveled, 12 Hour Time to Restart
3 Resinters, No Relevel, <1 Hour Time to Restart
3 Resinters, No Relevel, 4 Hour Time to Restart
3 Resinters, No Relevel, 12 Hour Time to Restart
3 Resinters, Releveled, <1 Hour Time to Restart
3 Resinters, Releveled, 4 Hour Time to Restart
3 Resinters, Releveled, 12 Hour Time to Restart
AMS Minimums (UTS = 180 ksi, Elongation = 10%)

Results


Ultimate Tensile Strength (ksi)
Yield Strength (ksi)
0
20
40
60
80
100
120
140
160
180
200
220
1 Resinter, No Relevel, <1 Hour Time to Restart
1 Resinter, No Relevel, 4 Hour Time to Restart
1 Resinter, No Relevel, 12 Hour Time to Restart
1 Resinter, Releveled, <1 Hour Time to Restart
1 Resinter, Releveled, 4 Hour Time to Restart
1 Resinter, Releveled, 12 Hour Time to Restart
3 Resinters, No Relevel, <1 Hour Time to Restart
3 Resinters, No Relevel, 4 Hour Time to Restart
3 Resinters, No Relevel, 12 Hour Time to Restart
3 Resinters, Releveled, <1 Hour Time to Restart
3 Resinters, Releveled, 4 Hour Time to Restart
3 Resinters, Releveled, 12 Hour Time to Restart
AMS Minimums (UTS = 180 ksi, Elongation = 10%)

Results

- As the AMS minimums are not sufficient for SLM material, the following acceptance criteria were chosen based on past testing experience:
  - Average Ultimate Tensile Strength: greater than 197 ksi.
  - Average Yield Strength: greater than 167 ksi.
  - Average Fracture Elongation: greater than 20%.
- The average of the three specimens per run was calculated and compared to the above acceptance criteria. Runs that fail are highlighted red. Additionally, if at least one individual specimen fails the above criteria, but the run average passes, that value is highlighted yellow.
- Note that a more robust, statistically based acceptance criteria and certification process is in development for SLM produced material. The acceptance criteria used herein should be considered as a "rule of thumb" based on tensile properties observed from specimens produced using the M1 SLM machine with this heat treatment process, and are subject to revision as new data is produced.

Run Number	Number of Re-sinters	Re-level Required	Time to Restart (hours)	Average Tensile Strength (ksi)	Average Yield Strength (ksi)	Average Fracture Elongation (%)	Std. Dev. Tensile Strength (ksi)	Std. Dev. Yield Strength (ksi)	Std. Dev. Fracture Elongation (%)
1	1	No	< 1	170.77	168.89	1.00	11.42	4.70	0.37
2	1	No	4	108.54	---	0.41	33.12	--	0.11
3	1	No	12	204.23	172.05	20.98	0.93	0.49	1.55
4	1	Yes	< 1	193.82	164.72	18.25	4.06	2.78	3.00
5	1	Yes	4	194.59	165.58	16.49	3.64	1.83	3.56
6	1	Yes	12	202.97	171.80	21.18	0.78	0.71	0.29
7	3	No	< 1	203.78	171.17	21.69	0.81	0.86	0.72
8	3	No	4	192.85	166.96	10.40	1.84	0.31	2.21
9	3	No	12	194.52	164.89	19.45	2.81	1.44	1.88
10	3	Yes	< 1	203.54	171.54	22.34	0.50	0.82	0.51
11	3	Yes	4	202.67	170.57	21.66	0.52	0.45	0.60
12	3	Yes	12	202.39	169.56	22.22	2.08	1.20	1.68
Control	---	---	---	200.69	167.00	21.02	0.72	0.63	0.50

= Mean fails minimum

= Sample fails minimum

Results

- For runs with a single re-sinter (Runs 1 through 6), only the 12 hour restart time specimens yielded acceptable properties, with the exception of one specimen with slightly lower elongation.
- For runs requiring a re-level (Runs 4, 5, 6, 10, 11, and 12), all of the runs that were re-sintered 3 times passed the acceptance criteria, regardless of restart time. Only the 12 hour restart time passed the acceptance criteria for runs that were re-sintered once.

Run Number	Number of Re-sinters	Re-level Required	Time to Restart (hours)	Average Tensile Strength (ksi)	Average Yield Strength (ksi)	Average Fracture Elongation (%)	Std. Dev. Tensile Strength (ksi)	Std. Dev. Yield Strength (ksi)	Std. Dev. Fracture Elongation (%)
1	1	No	< 1	170.77	168.89	1.00	11.42	4.70	0.37
2	1	No	4	108.54	---	0.41	33.12	--	0.11
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12	3	Yes	12	202.39	169.56	22.22	2.08	1.20	1.68
Control	---	---	---	200.69	167.00	21.02	0.72	0.63	0.50

= Mean fails minimum

= Sample fails minimum

Results

- For 3 re-sinters, failure seems to be related to re-leveling, and the shorter times exhibit better properties.
- At the beginning of this study, the intuition was that re-leveling would be detrimental; however, for 3 re-sinters, a re-level is better.
- In all cases, 4 hours of restart time is the worst in terms of elongation, and is worst in tensile strength in all but one case.
- Results indicate that 3 re-sinters and a re-level, regardless of whether Z height was lost or not, should be considered as a potential practice moving forward.
- Again, an alternative acceptance criteria might produce different conclusions.

Run Number	Number of Re-sinters	Re-level Required	Time to Restart (hours)	Average Tensile Strength (ksi)	Average Yield Strength (ksi)	Average Fracture Elongation (%)	Std. Dev. Tensile Strength (ksi)	Std. Dev. Yield Strength (ksi)	Std. Dev. Fracture Elongation (%)
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6	1	Yes	12	202.97	171.80	21.18	0.78	0.71	0.29
7	3	No	< 1	203.78	171.17	21.69	0.81	0.86	0.72
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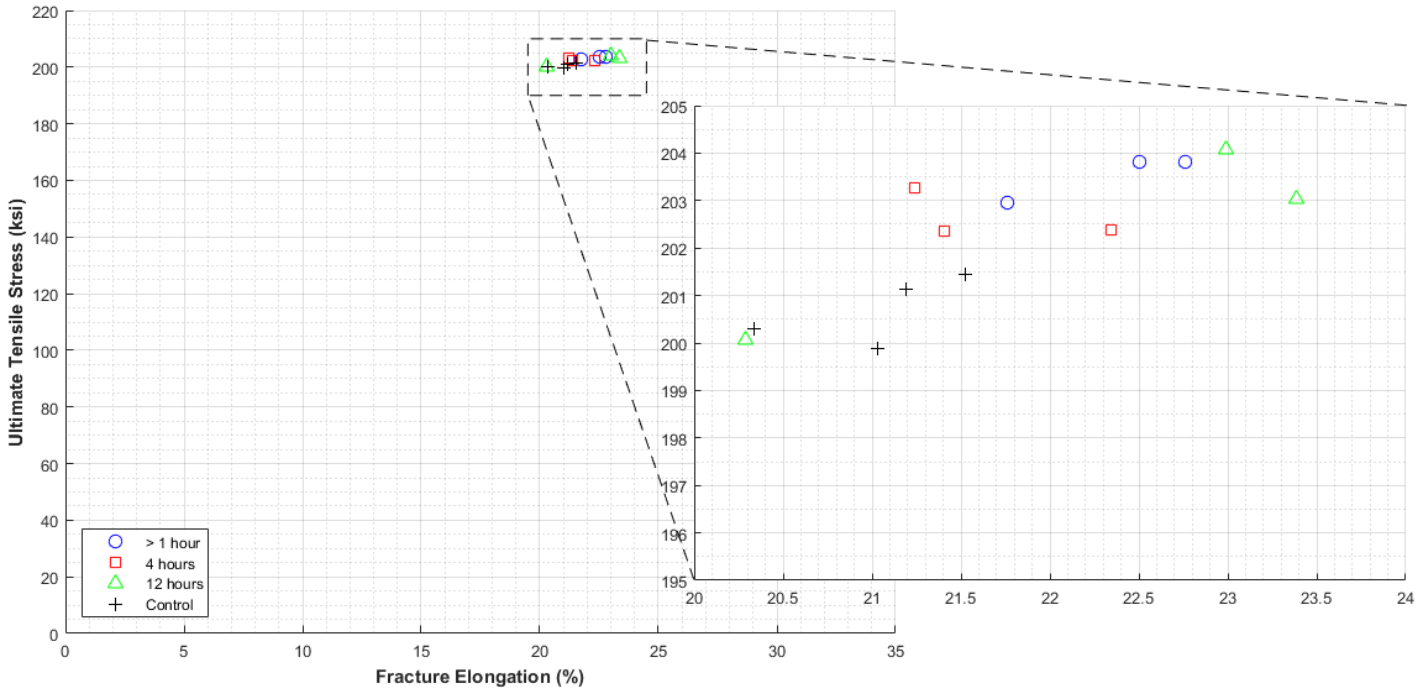
10	3	Yes	< 1	203.54	171.54	22.34	0.50	0.82	0.51
11	3	Yes	4	202.67	170.57	21.66	0.52	0.45	0.60
12	3	Yes	12	202.39	169.56	22.22	2.08	1.20	1.68
Control	---	---	---	200.69	167.00	21.02	0.72	0.63	0.50

= Mean fails minimum

= Sample fails minimum

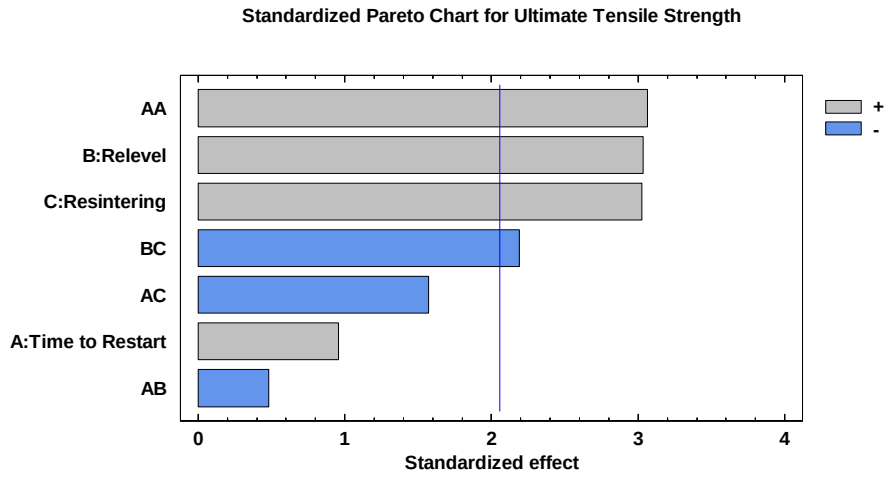
Results – Re-leveled and 3 Re-sinters

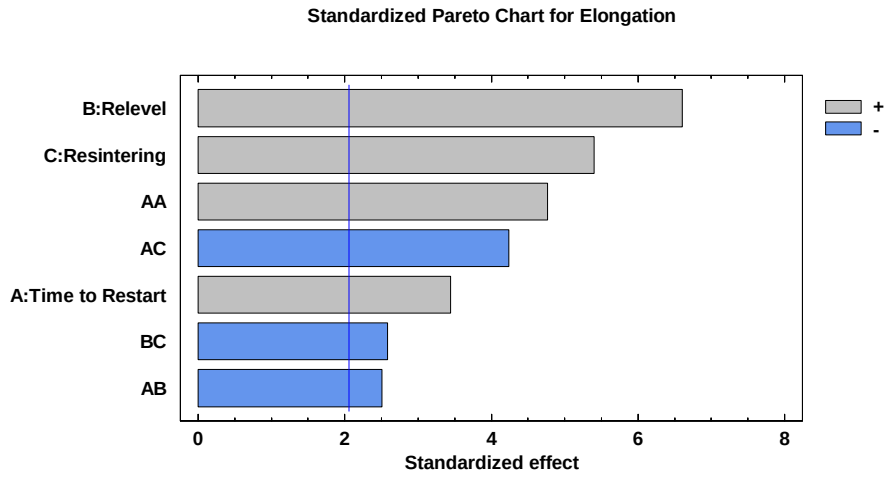
- The results for re-leveled and 3 re-sinter specimens are shown here.
- Results are all within family.
- The control specimens with no restarts are plotted as well. The control specimens are a different specimen design, and have been shown to result in slightly lower strength and elongation results. See Test Report #2015-0537 Seq 4 for details.
- The test results from this grouping indicated acceptable properties that are in family with the control specimens.



Results – Statistical Analysis

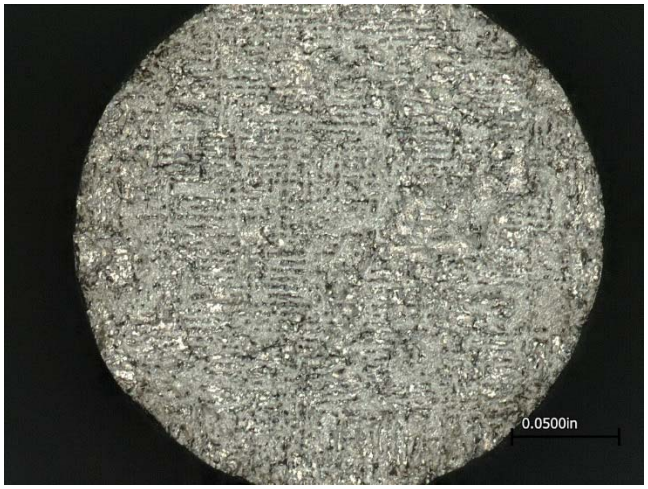
- A statistical DOE analysis was performed on the test results using Statgraphics.
- The Pareto Charts shown below indicate the relative importance of the tested factors. Factors that exceed the vertical blue construction line are considered statistically significant.
- For both ultimate tensile strength and total elongation, re-leveling and re-sintering are highly important, indicating that re-leveling and the number of re-sinters have a significant effect on the observed material properties.
- Time to restart is a significant effect for elongation, although not as important as re-leveling and re-sintering, but is not as significant effect for ultimate tensile strength.



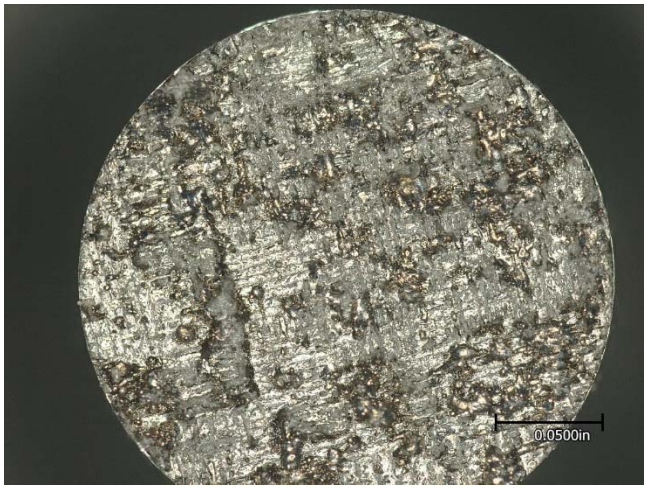


Fracture Surfaces – Representative of Runs 1 and 2

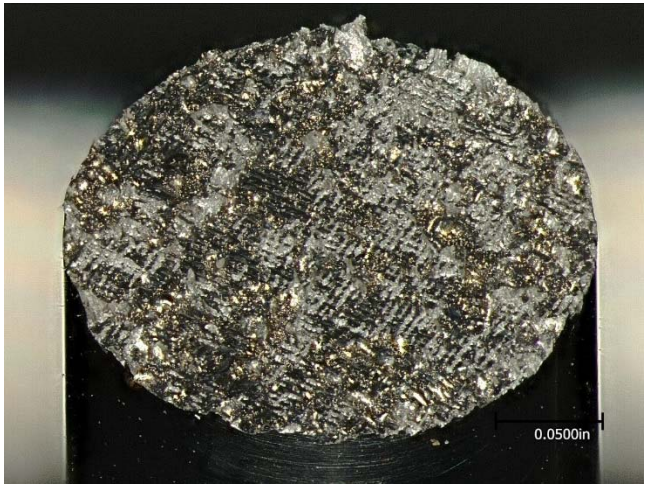
- Several observed specimen fracture surfaces exhibited evidence of incomplete sintering between layers at the restart. These specimens exhibited some of the worst material properties.



11N1B - Fail



21N4A - Fail



21N4A - Fail

Fracture Surfaces – Representative of Runs 10 and 12

- Other fracture surfaces exhibited character that would be expected from SLM produced material. These specimens were above the material property acceptance criteria.

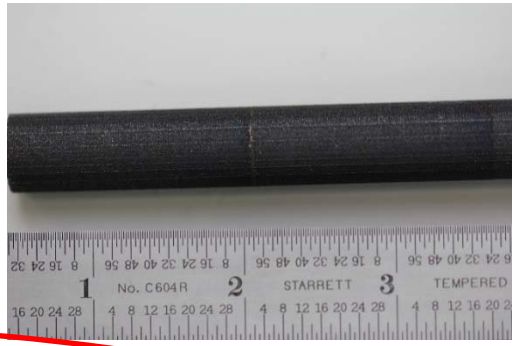
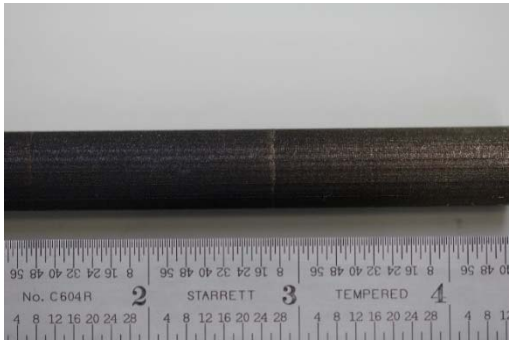
103Y1A - Pass

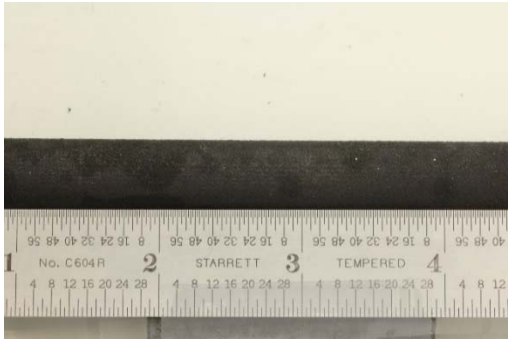
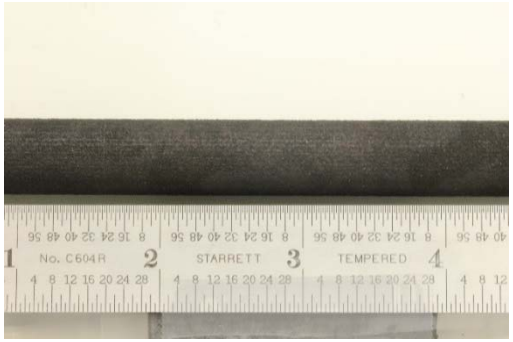
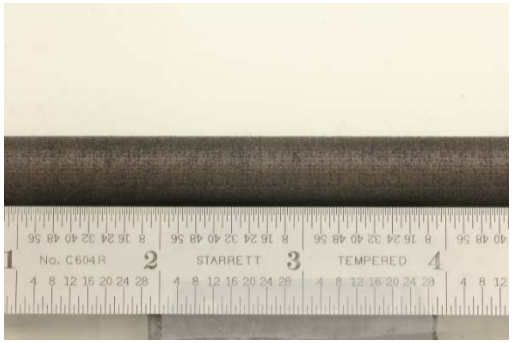
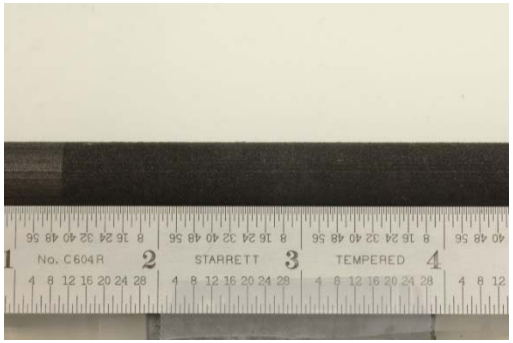
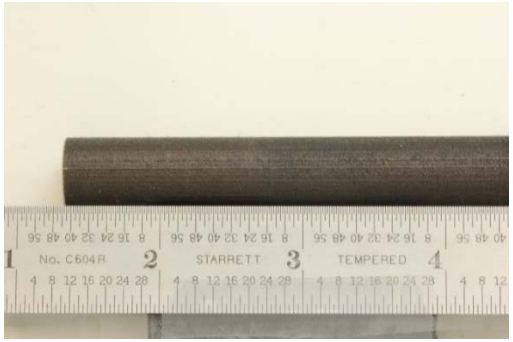
123Y12A - Pass

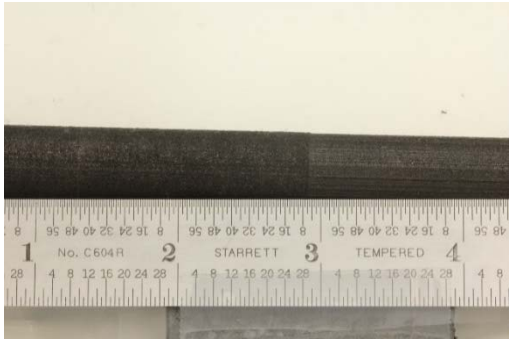
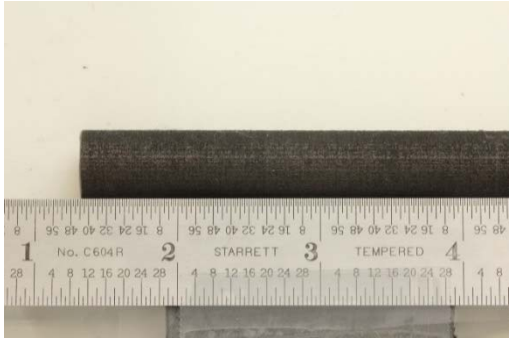
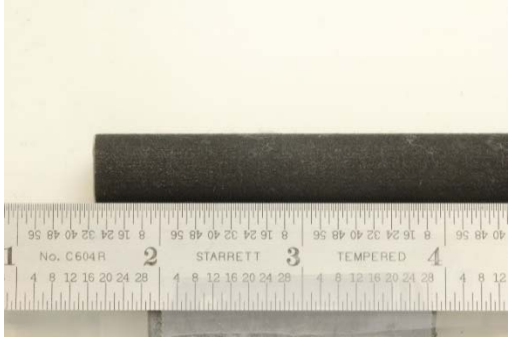
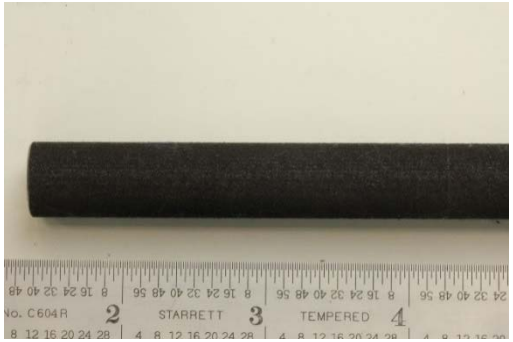
123Y12A - Pass

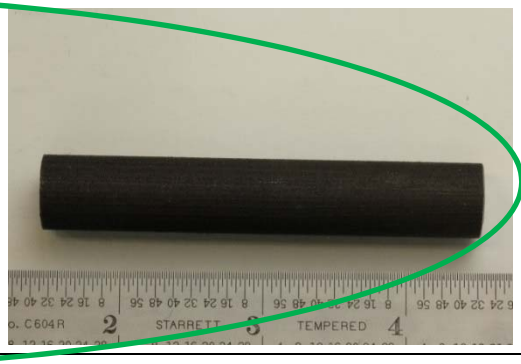
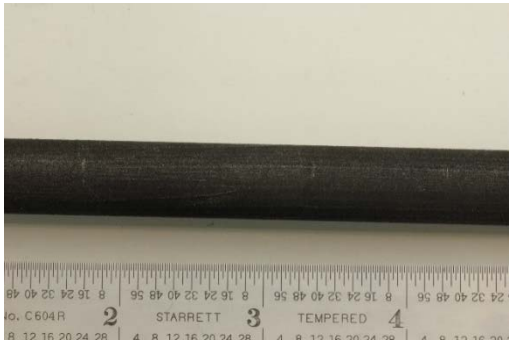
Results – Visual Inspection

- The following charts show images of the restart lines in the specimens, post-stress relief.
- Many of the restart lines are easily detectable with a visual inspection.
- Qualitatively, the visibility of the restart lines loosely correlates with the resulting material properties. Restart lines that are easier to see tend to result in worse material properties.

	Specimen A	Specimen B	Specimen C
Run 1: 1 Re-sinter, No Re-level, <1 hours Fail			
Worst Properties Run 2: 1 Re-sinter, No Re-level, 4 hours Fail			
Run 3: 1 Re-sinter, No Re-level, 12 hours Borderline			

	Specimen A	Specimen B	Specimen C
Run 4: 1 Re-sinter, Yes Re-level, <1 hours Fail			
Run 5: 1 Re-sinter, Yes Re-level, 4 hours Fail			
Run 6: 1 Re-sinter, Yes Re-level, 12 hours Pass			

	Specimen A	Specimen B	Specimen C
Run 7: 3 Re-sinters, No Re-level, <1 hours Pass			
Run 8: 3 Re-sinters, No Re-level, 4 hours Fail			
Run 9: 3 Re-sinters, No Re-level, 12 hours Fail			

	Specimen A	Specimen B	Specimen C
Best Properties Run 10: 3 Re-sinters, Yes Re-level, <1 hours Pass			
Run 11: 3 Re-sinters, Yes Re-level, 4 hours Pass			
Run 12: 3 Re-sinters, Yes Re-level, 12 hours Pass			

Restart Images – Best and Worst

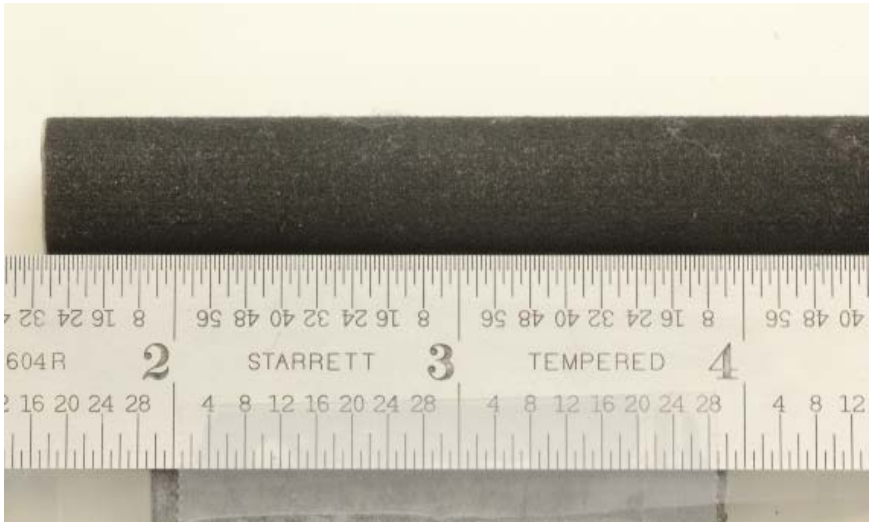
- Images of the test specimens with the worst properties and the best properties are shown here.
- The restart line is more noticeable on the poorly performing specimen than the best specimen. In general, the worst specimens had restart lines that were apparent upon visual examination.

21N1C - Fail

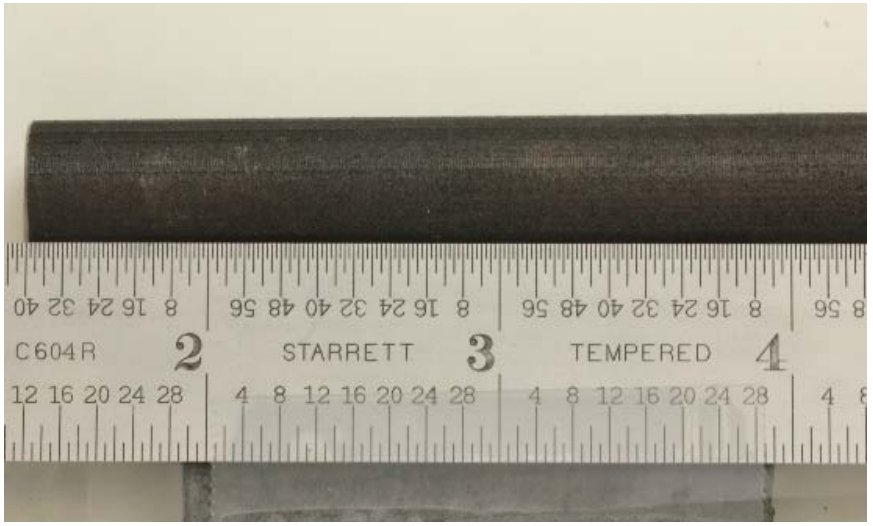
103Y1A - Pass

Restart Images – Best of the Fails/ Worst of the Passes

- However, in a few cases, a restart line that was difficult to see resulted in poor properties. In these images, both lines are difficult to observe, but resulted in different material properties upon testing.
- All of the specimens that produced passing properties contained restart lines that were relatively difficult to observe.



93N12B - Fail



61Y12B - Pass

Summary and Future Reporting Observations:

Summary

- Results indicate that re-leveling and re-sintering three times produces the best material properties. There does not seem to be a strong effect of time to restart on the properties of the specimens that were both re-leveled and re-sintered three times.
- Statistical analyses indicate that re-leveling and re-sintering, and in some cases time to restart, are significant effects that does have a meaningful impact on the observed material properties.
- In general, material properties loosely correlated with the visibility of the restart lines.

Recommendations

- Further studies that would enhance the conclusions of this study include:
    - Determine NDE detectability of problematic restart lines.
    - Further investigate the time to restart effect on restarts with a re-level and three re-sinters.
    - Compare re-leveled and three re-sinter specimens to specimens without restart lines.

Appendices

Heat Treatment Specifications

- Stress Relief:
  - 1950°F ± 25°F for 1.5 hrs. -5/+15 min., 2-4 Bar argon quench.

- AMS 5664E:
  - *Solution heat treatment*: Using a ramp rate of approximately 25°F per minute, solution treat in vacuum at 1925°F ±10°F for 1 hour, followed by an in-furnace argon quench.

  - *Precipitation heat treatment*: Age in vacuum at 1400°F ± 15°F for 10 hrs. ±0.5 hrs., furnace cooling to 1200°F ± 10°F, holding at 1200°F ±10°F until a total precipitation heat treatment time of 20 hrs. has been reached, and cooled.