Minimizing Titanium Alpha Case During Vacuum Furnace Heat Treating

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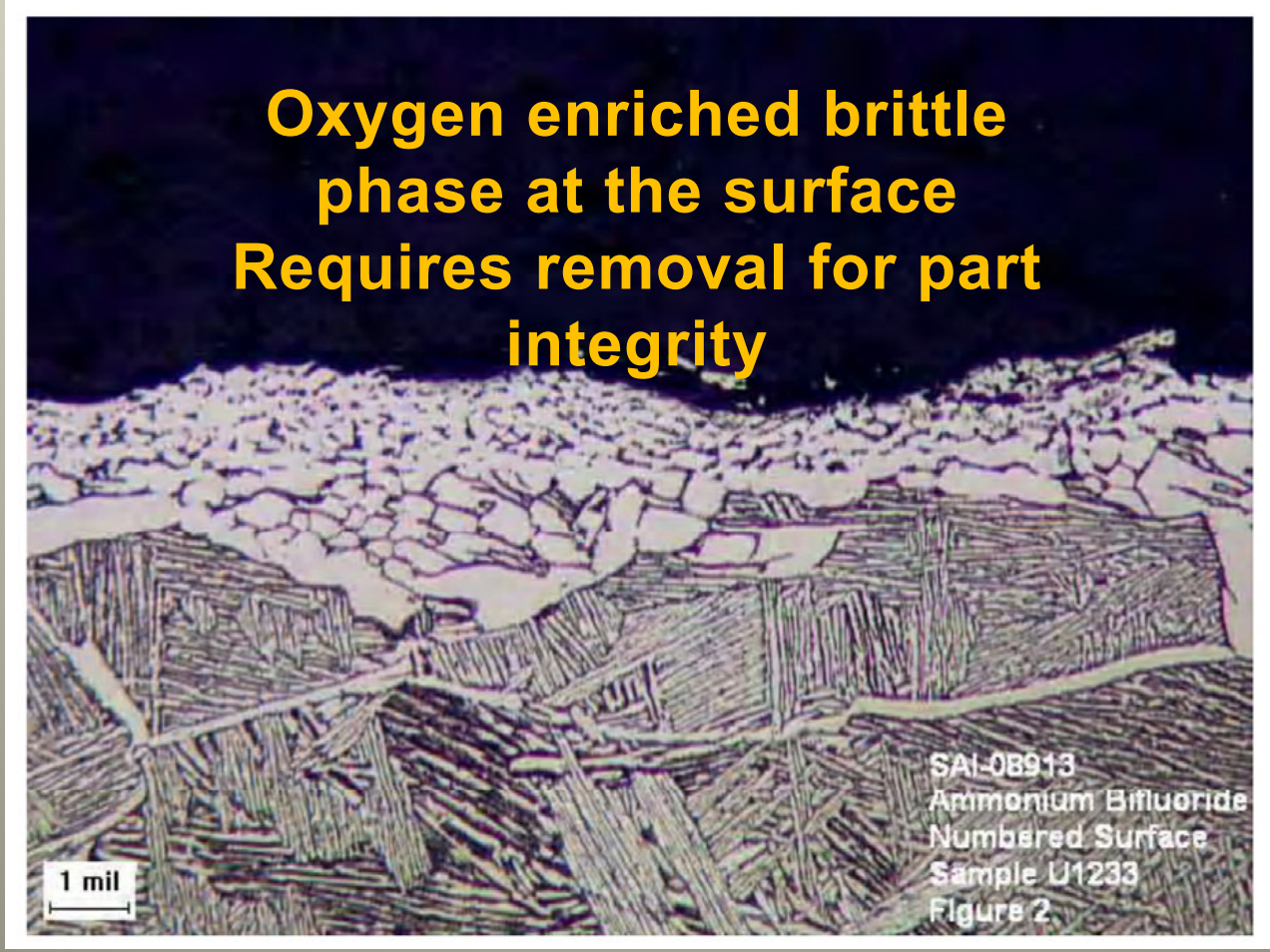
Overview

- Titanium alpha case forms at elevated temperatures principally due to oxygen as revealed by a residual gas analyzer
- Color has been used as a post welding and heat treating criterion for the presence of alpha case
- Do visible color hues relate to the extent of alpha case depths?
- Hypothesis - Spectrophotometer can be used to indicate the extent of alpha case
- Evaluate etchants used in metallographic analysis of alpha case

What is Alpha Case?



Titanium Color as an Oxide Film

Table 2—Color Acceptance Criteria

Weld Color	Quality Indication
Bright Silver	Acceptable[a]
Silver	Acceptable[a]
Light Straw	Acceptable[a]
Dark Straw	Acceptable[a]
Bronze	Acceptable[a]
Brown	Acceptable[a]
Violet	Unacceptable[b, c]
Dark Blue	Unacceptable[b, c]
Light Blue	Unacceptable[b, c]
Green	Unacceptable[b, c]
Gray	Unacceptable
White	Unacceptable

http://www.millerwelds.com/resources/articles/TIG-gtaw-titanium-welding

Oxide appearing bright/white

Different colors are exhibited owing to a change in the thickness of the oxide layer.

http://www.rockymountainlabs.com/newsletters/Layer%20Thickness%20-

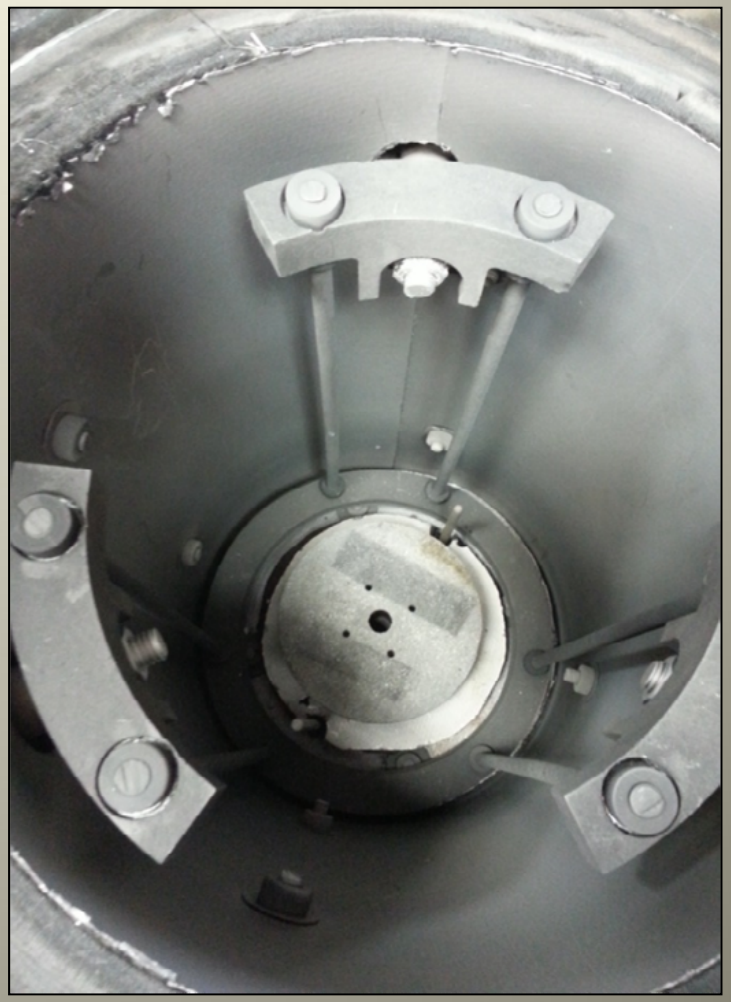
Sound Practice For Alpha Case Prevention in Vacuum

- Bake out furnace in deep vacuum at 100° or higher than process hold temperature
- Pump to $1X10^{-4}$ Torr or below
  - AMS2769 specifies 0.1 micron or lower
- Ramp relatively slowly: 600°F/hour
- If out-gassing occurs, hold at temperature until vacuum drops to $2X10^{-4}$ Torr

Experimental Design

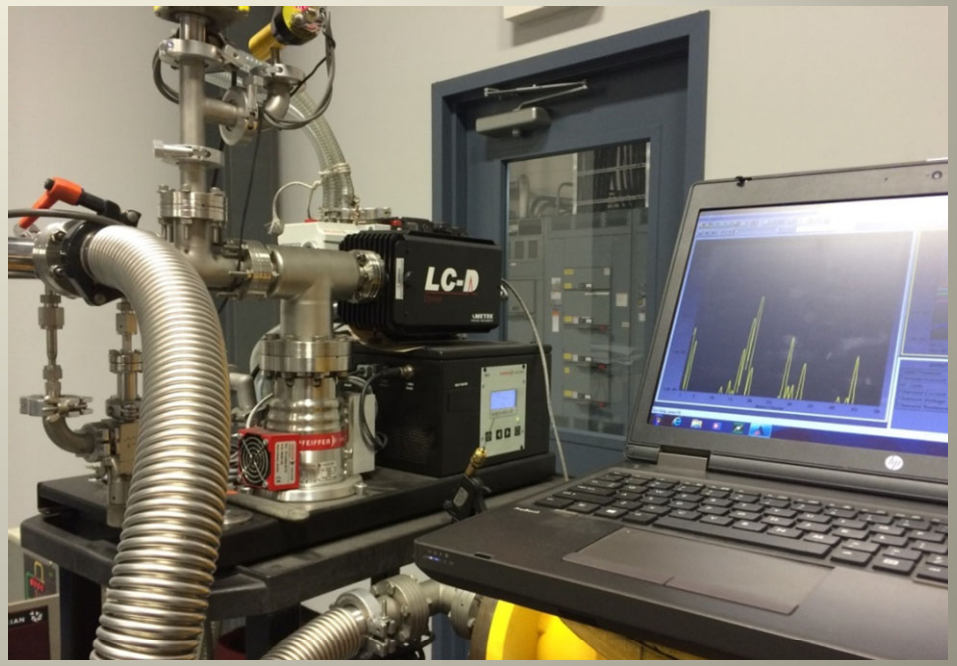
- Process in a baked-out all graphite insulated hot zone
- Use Residual Gas Analyzer (RGA) as an evaluation tool during processing
- Look at the effect of ramp rate and temperature on the extent of alpha case formation
- Investigate the effect of low temperature holds during ramp-up
- Determine if visible color hues relate to the extent of alpha case depths

Furnace Used for Testing

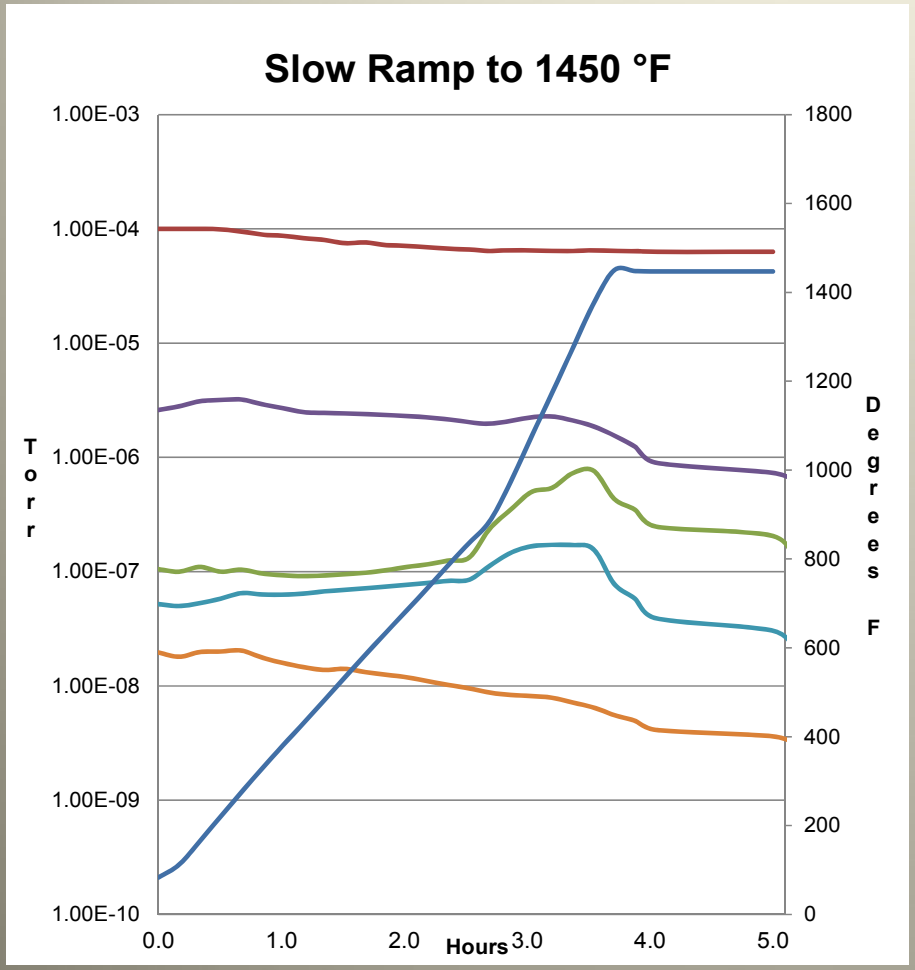


Experimental Parameters

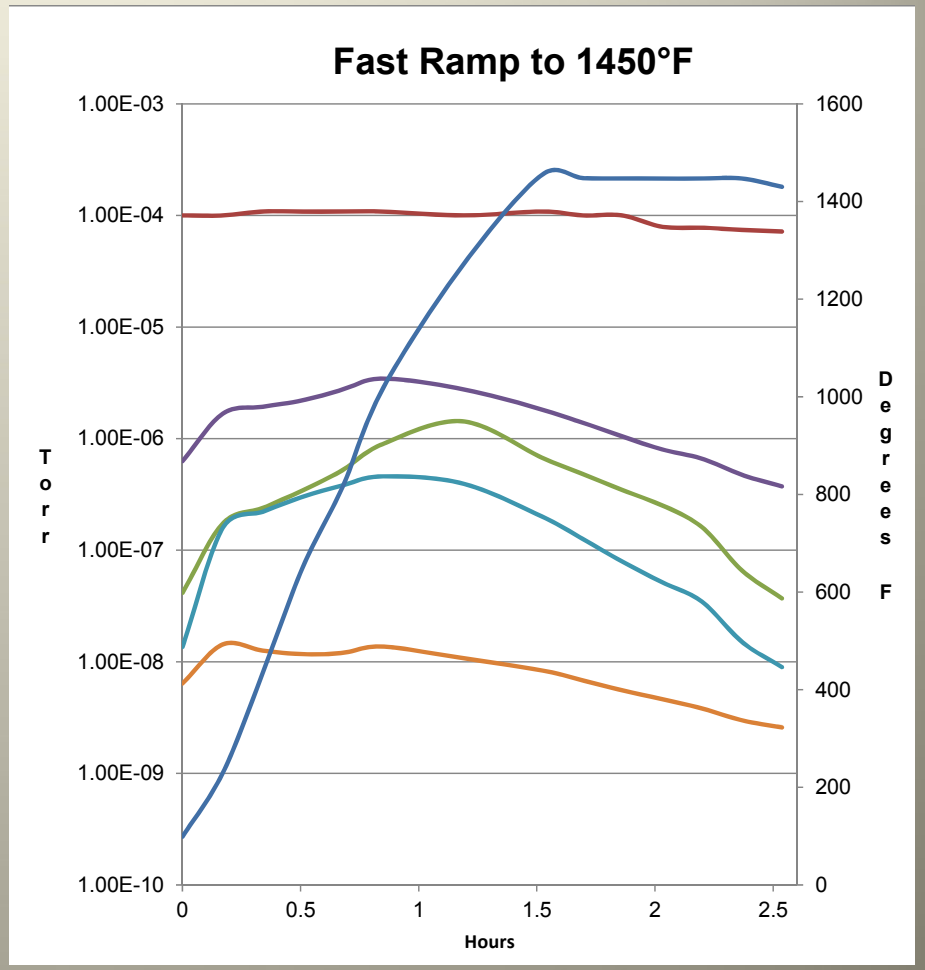
- Start all processes at 1 X10^{-4} Torr
- Two set temperatures:
  - 1450°F
  - 1750 °F
- Two ramp profiles:
  - 300°F/hour to 900°F
    600°F/hour to hold temperature
  - 1200°F/hour to hold temperature
- 1 hour hold time at set temperature
- Monitor heat cycle with RGA



RGA Ramp Rate Comparison



300°F/hour to 900°F
600°F/hour to 1450°F
1 hour hold

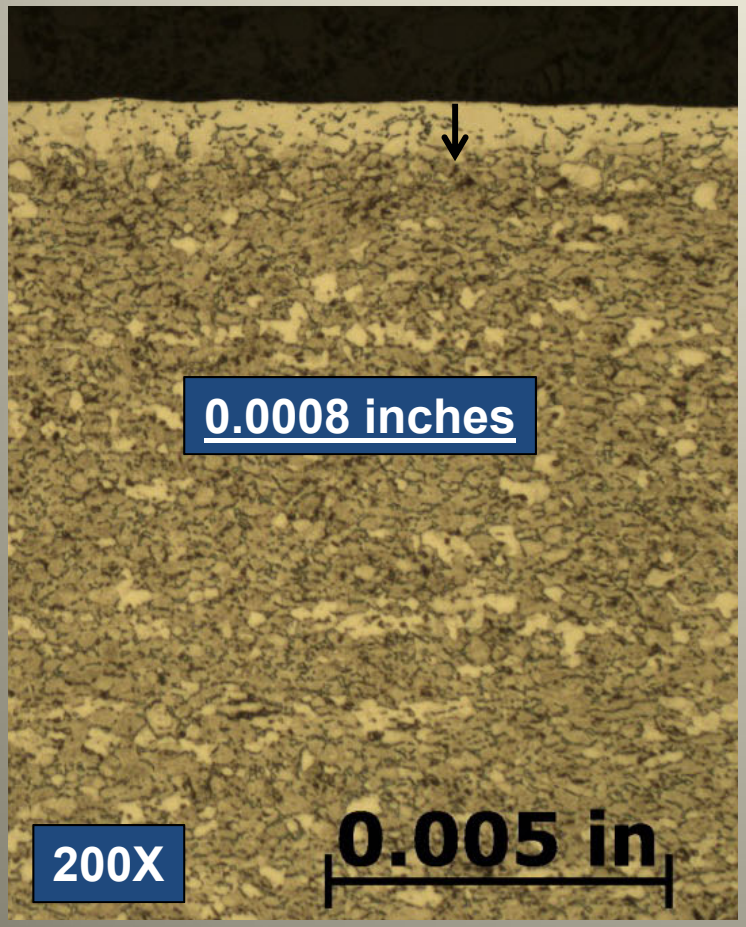


1200°F/hour to 1450°F
1 hour hold

Ramp Rate Did Not Influence Alpha Case

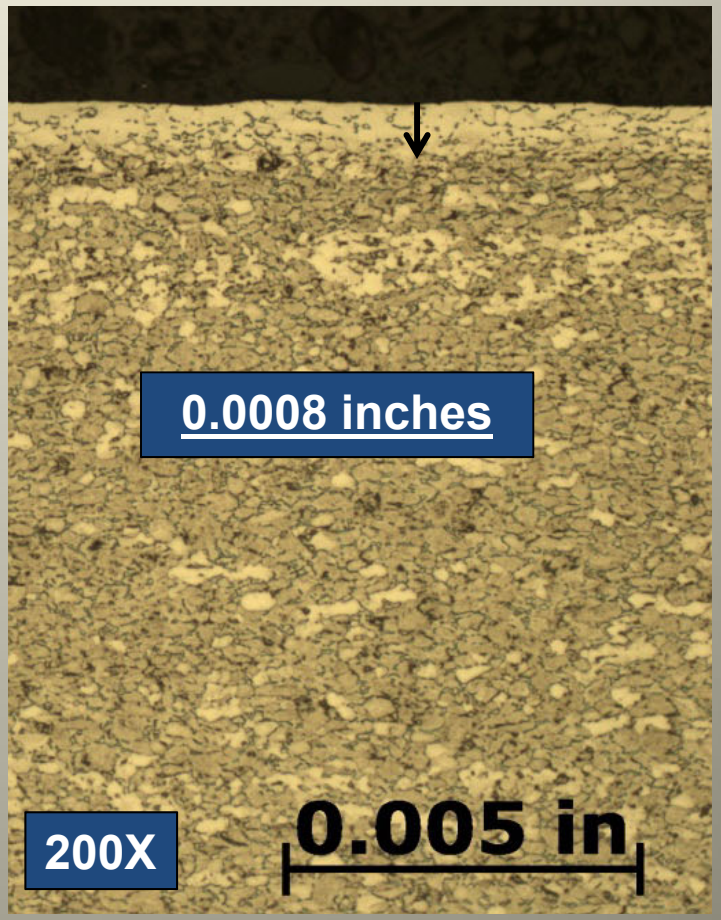
Slow Ramp to 1450°F

300°F/hour to 900°F
600°F/hour to 1450°F; 1 hour hold

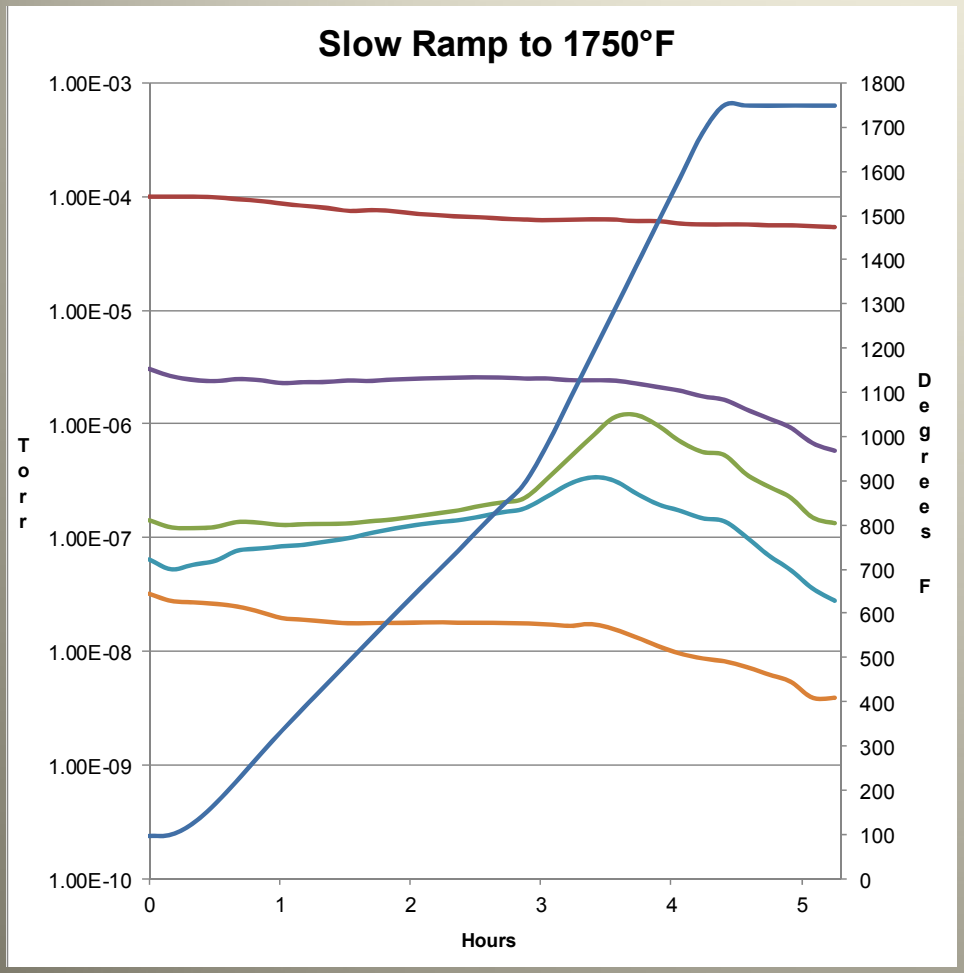


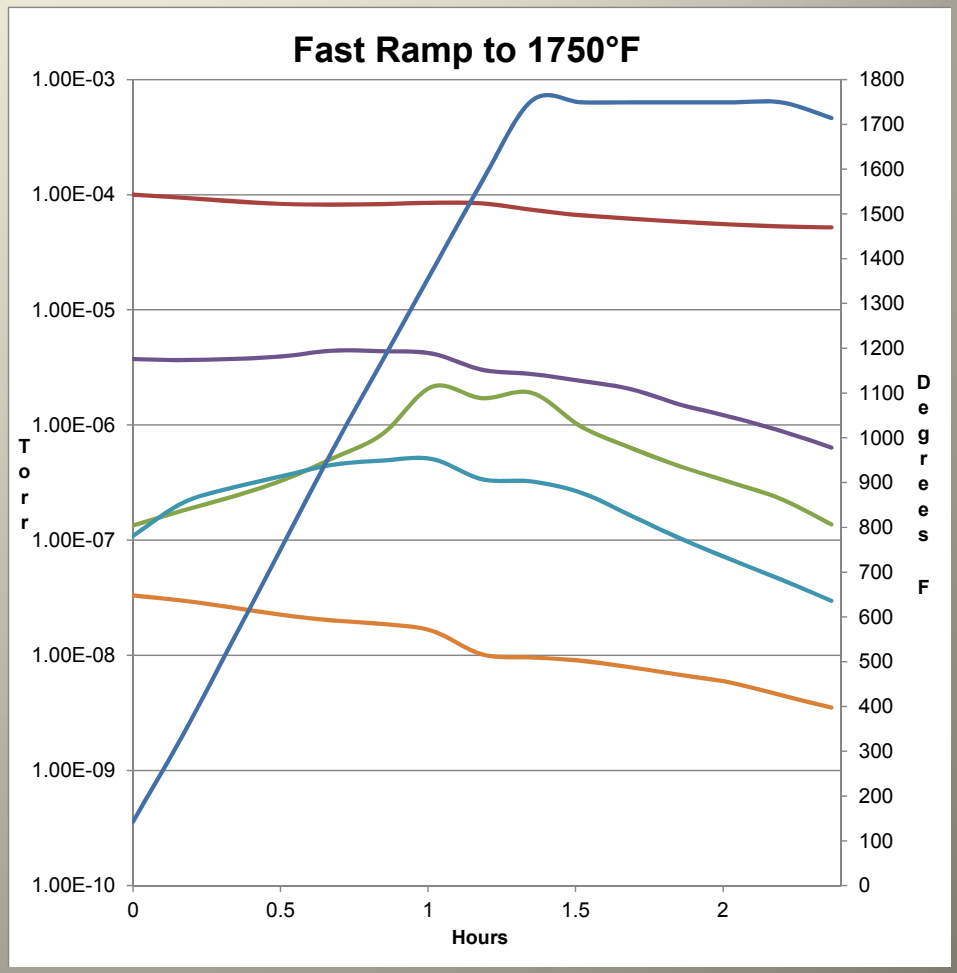
Fast Ramp to 1450°F

1200°F/hour to 1450°F; 1 hour hold



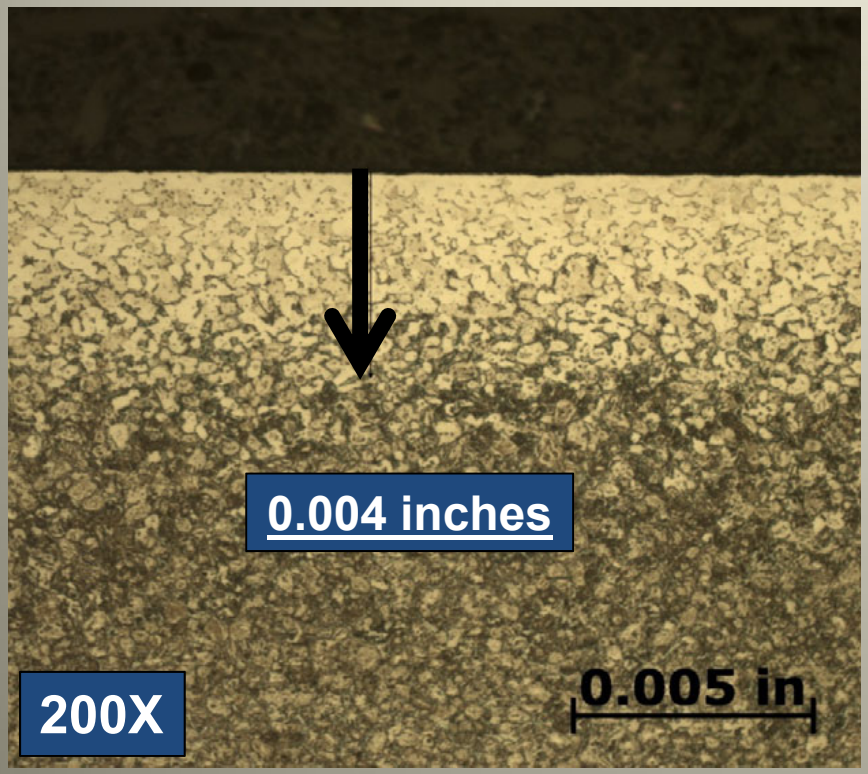
RGA Ramp Rate Comparison at 1750°F



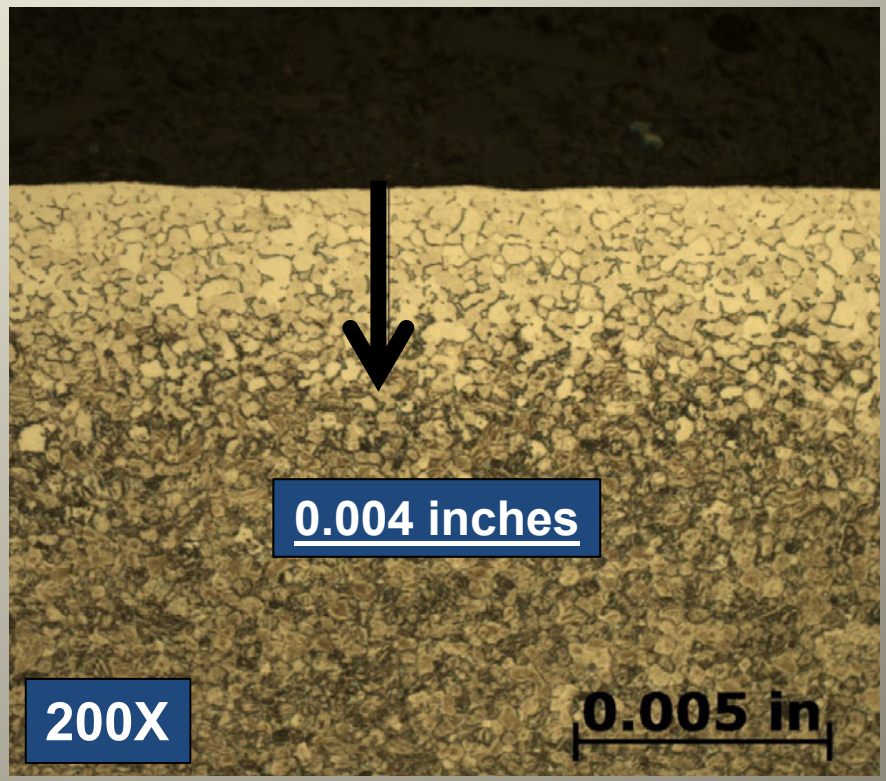


Ramp Rate Did Not Influence Alpha Case

Slow Ramp to 1750°F - Two Step

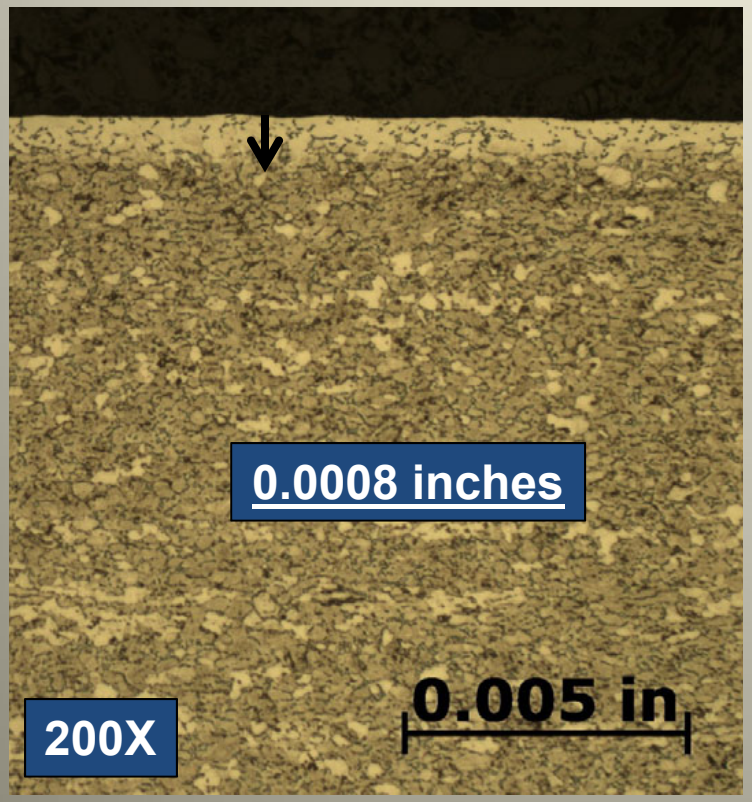


Fast Ramp to 1750°F – One Step

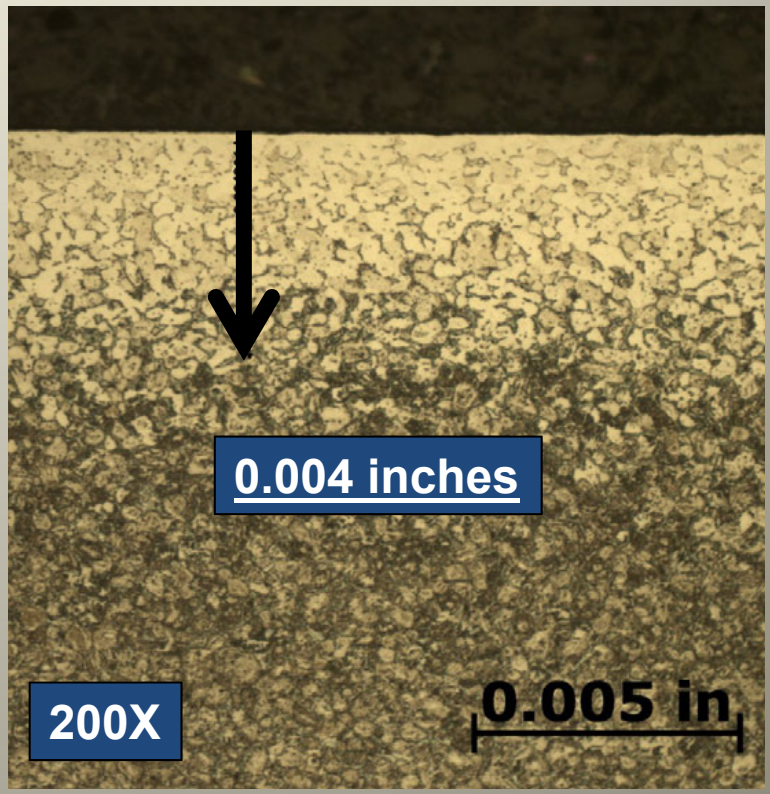


Final Temperature Greatly Influences Case Depth

1450°F



1750°F



Intermittent Holds Affect on Alpha Case

Current Theory:

- Introduce hold times during heat up to allow outgassing to occur at lower temperatures and reduce reactions on the heated surface

Test Cycle:

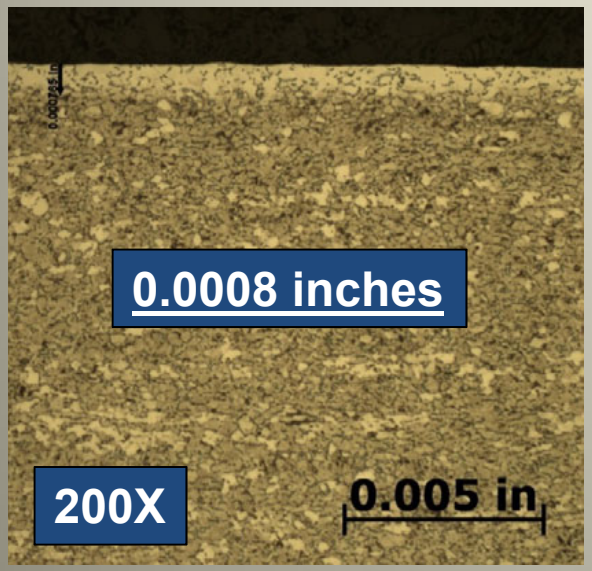
300 °F/hour to 600 °F; 1 hour hold

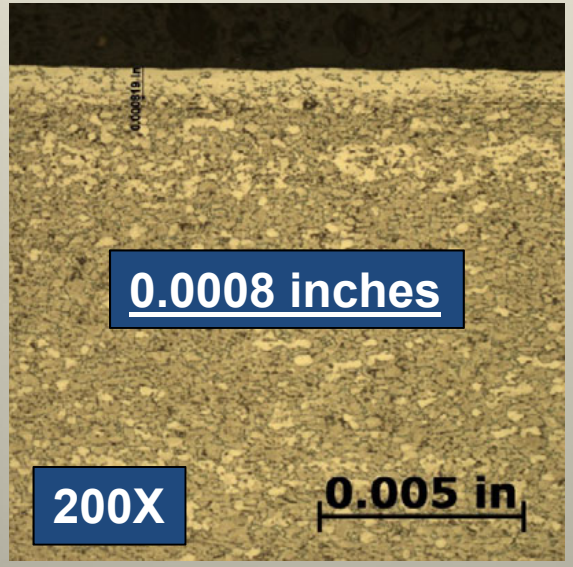
300 °F/hour to 900 °F; 1 hour hold

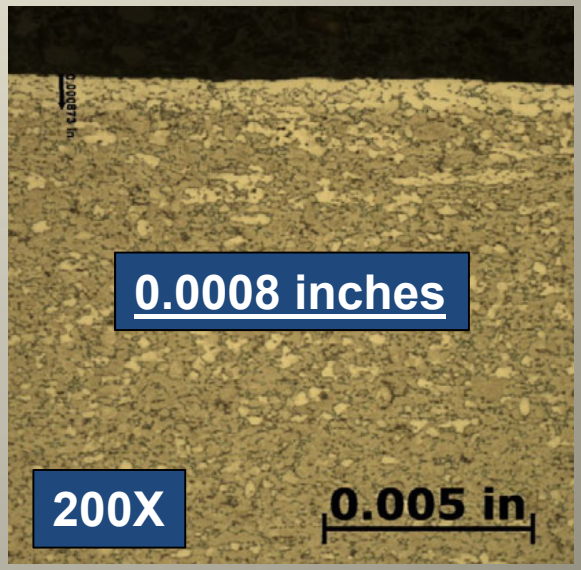
600 °F/hour to 1450 °F; 1 hour hold

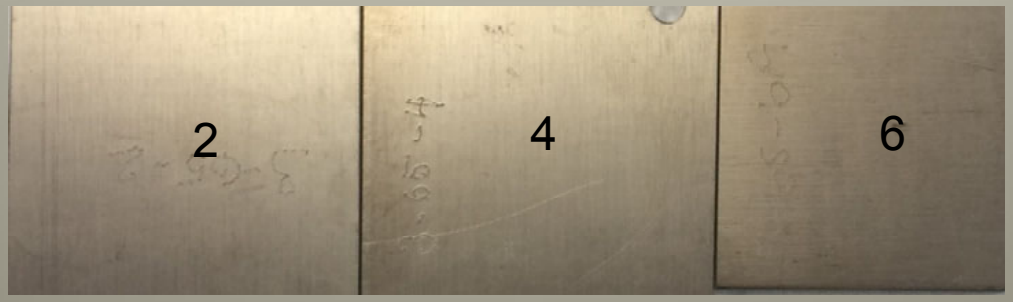
Slow Ramp Rate and Low Temperature Holds Did Not Minimize Alpha Case

Test 2	Test 4	Test 6
300°F/hour to 900°F 600°F/hour to 1450°F; 1 hour hold	1200°F/hour to 1450°F; 1 hour hold	300°F/hour to 600°F; 1 hour hold 300°F/hour to 900°F; 1 hour hold 600°F/hour to 1450°F; 1 hour hold









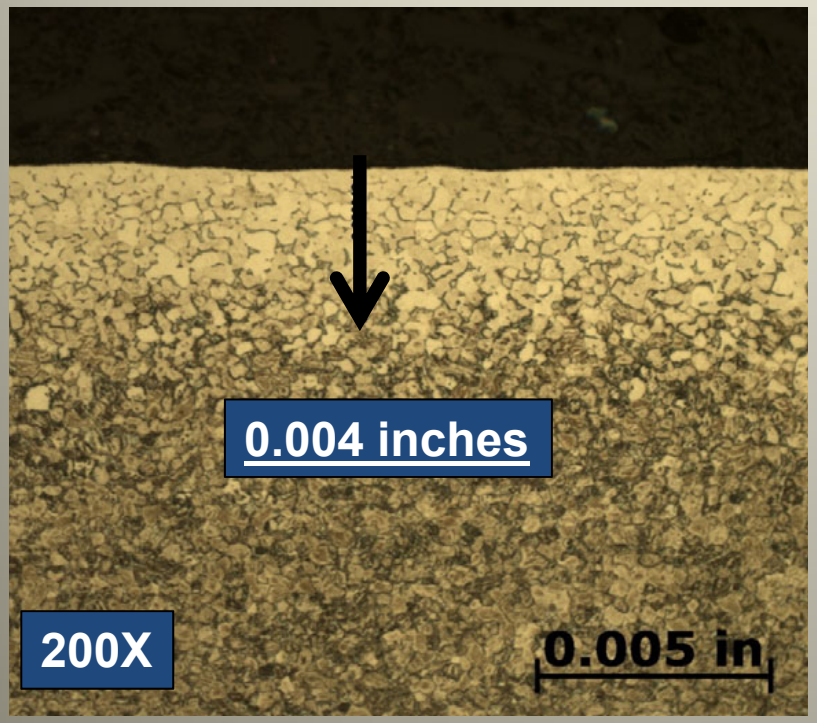
Heating Time:	5.2 hours	2.7 hours	7 hours

Affect of Surface Area on Alpha Case and Color

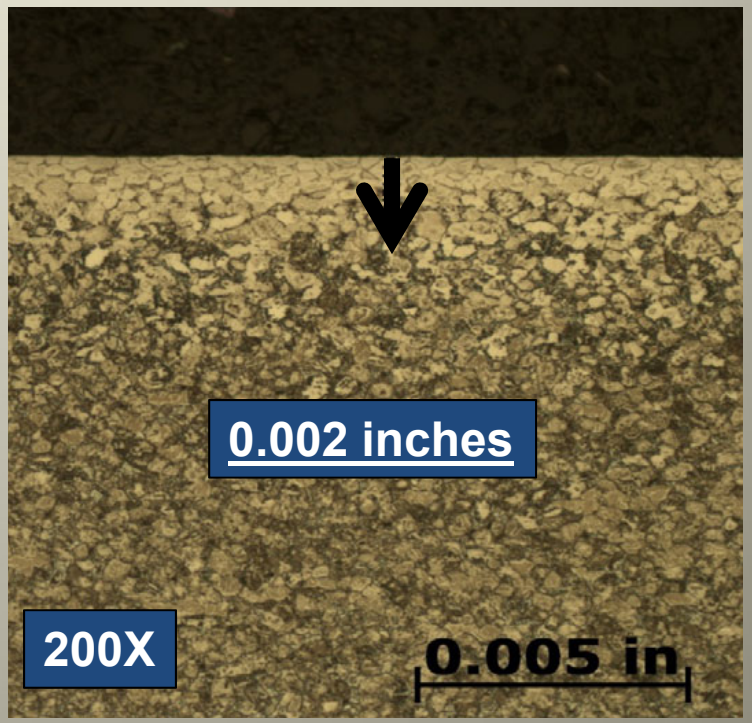
- Use several pieces of high purity Ti-6Al-4V parts
- Increased reactive surface area by factor of 9
- Ramp at 1200°F/hour to 1750°F; hold for 1 hour
- Compare alpha case depth and color to previous 1750°F test cycles

Increasing Surface Area Decreases Alpha Case

2 parts = 6.7 in^2

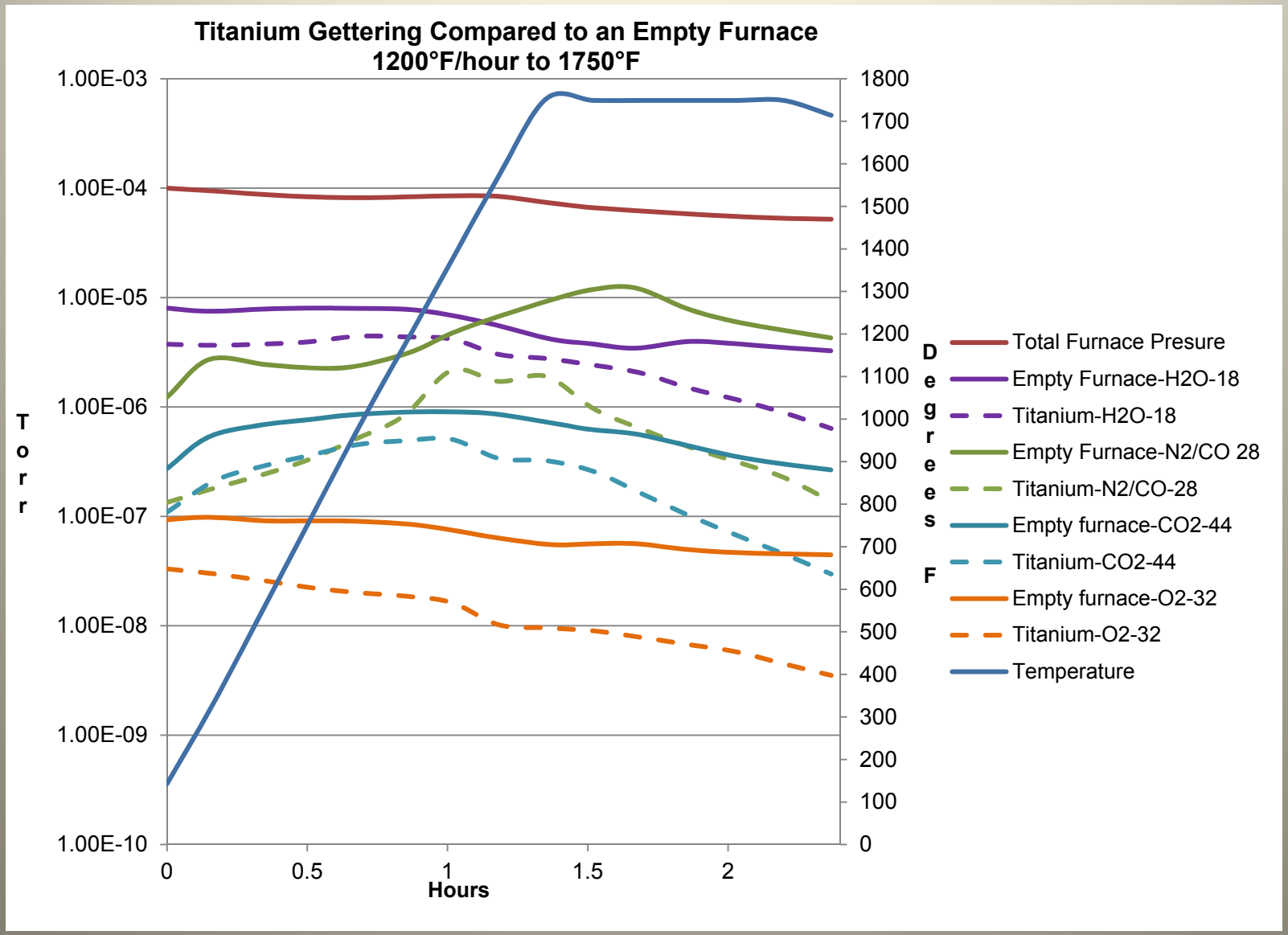


10 parts = 60 in^2

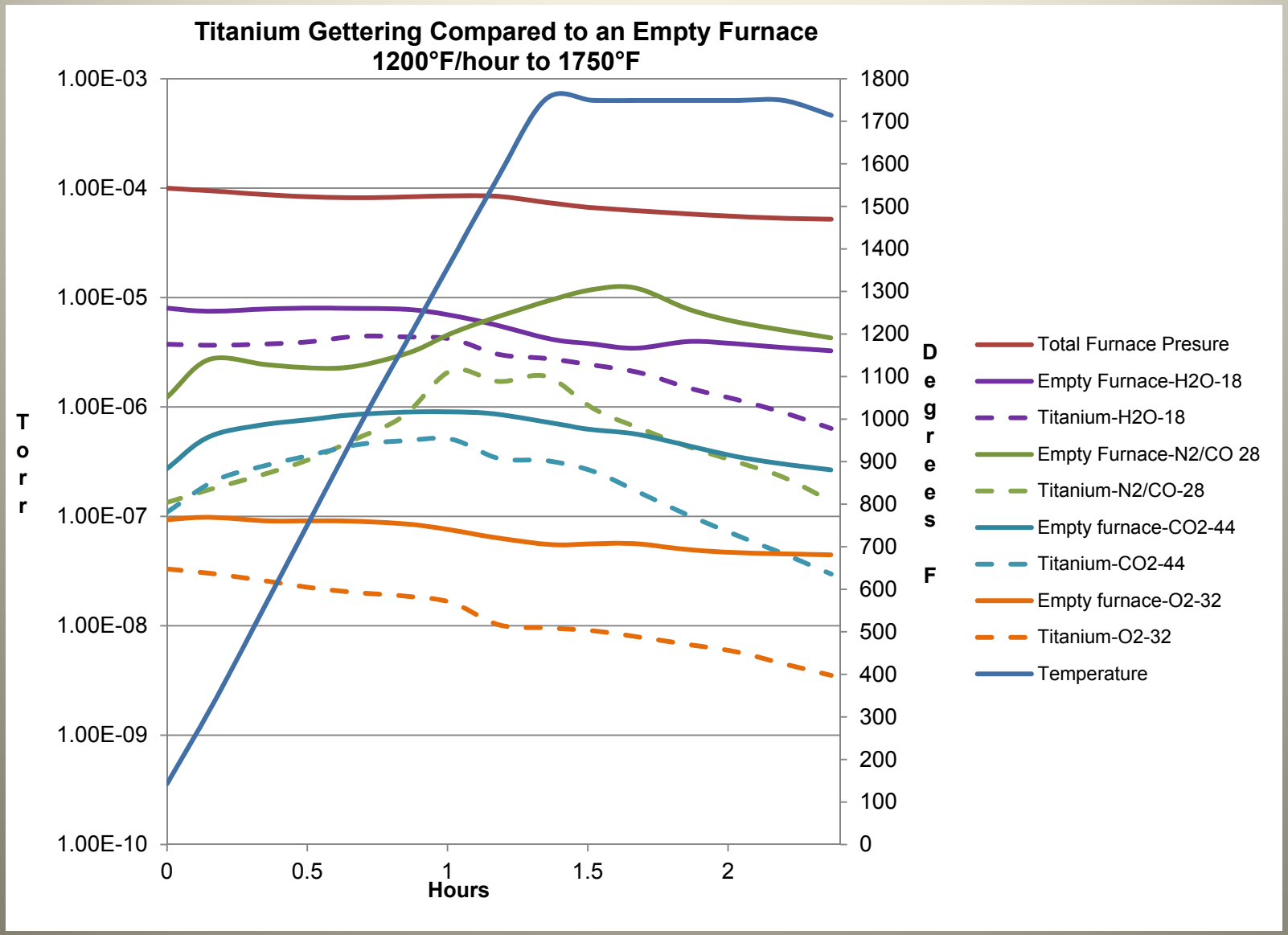


Surface Area increased by a factor of 9

Titanium Reacts with Residual Gases

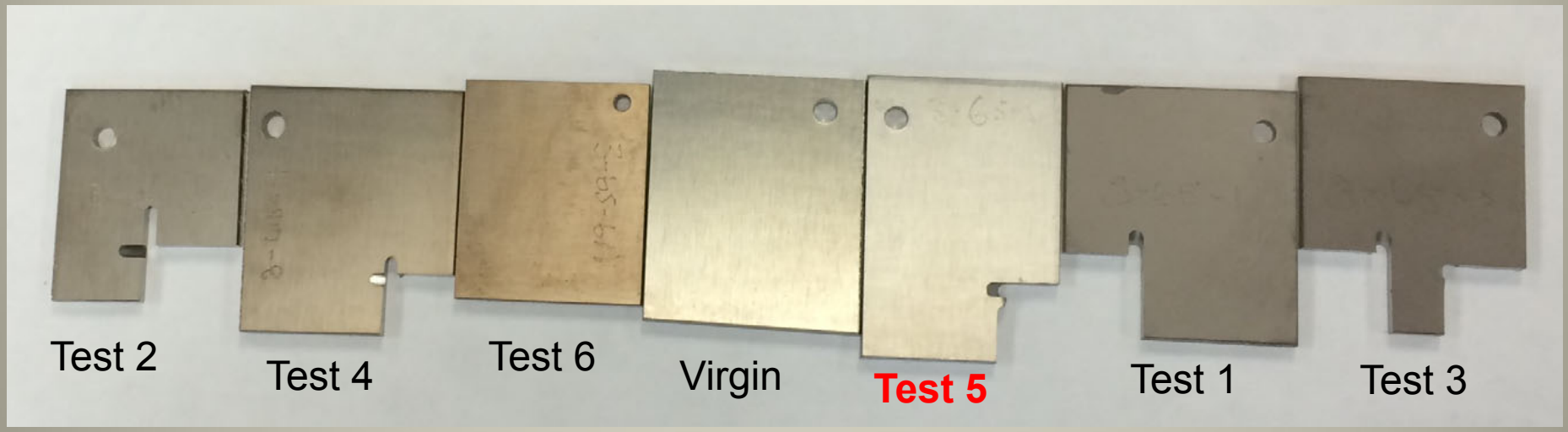


Titanium Reacts with Residual Gases



HunterLab Spectrophotometer

Appearance after Heat Treat vs Alpha Case

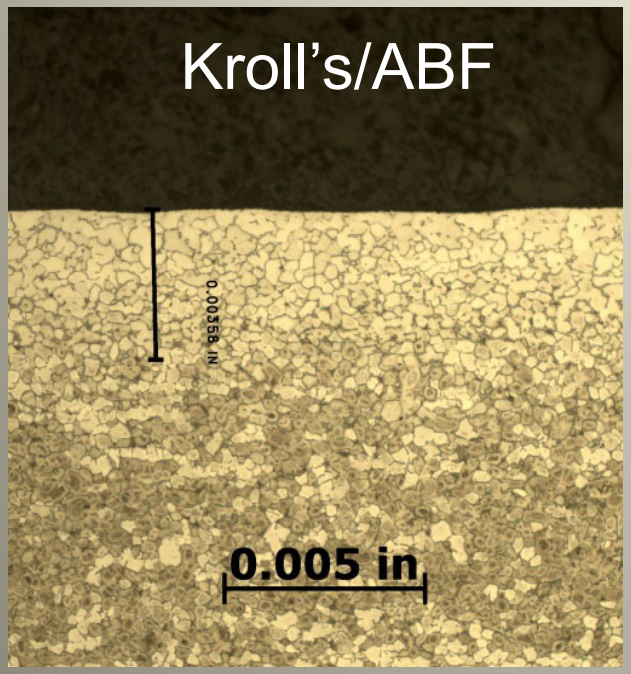


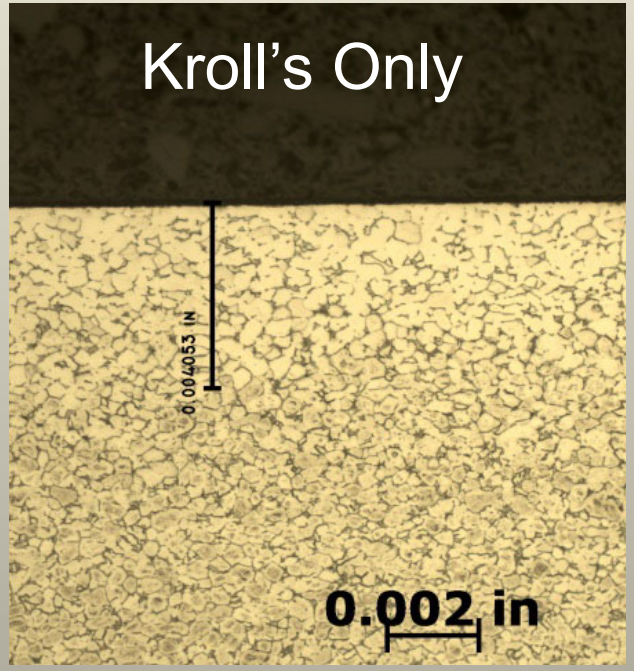
Test #	Final T (°F)	Ramp Type	Surface Area	Alpha Case (inches)	Appearance
1	1750	Two-step	1	0.004	Matte gray
2	1450	Two-step	1	0.0008	Yellow tint
3	1750	One-step	1	0.004	Matte gray
4	1450	One-step	1	0.0008	Yellow tint
5	1750	One-step	9	0.002	Bright
6	1450	Three-step	1	0.0008	Yellow tint
Virgin	NA	NA	NA	0.000	Bright

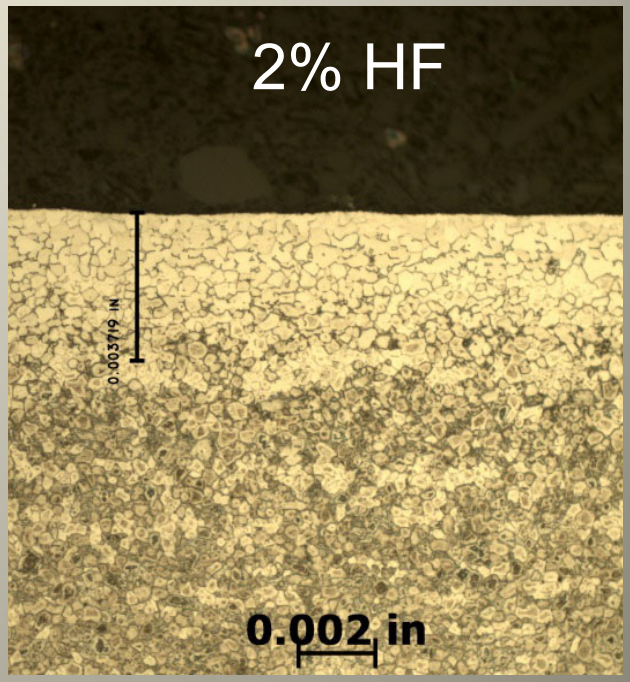
Metallography

- Etchant type and contact time influence the appearance of alpha case
- It was found that Kroll's followed by 2% Ammonium Bifluoride, and 2% HF both were effective for delineating alpha case
- HF alone was considered most effective because of ease of use compared to having perform two etching steps, but increased contact time can decrease the apparent case
- Kroll's alone is least effective in delineating alpha case

Etchant Comparison

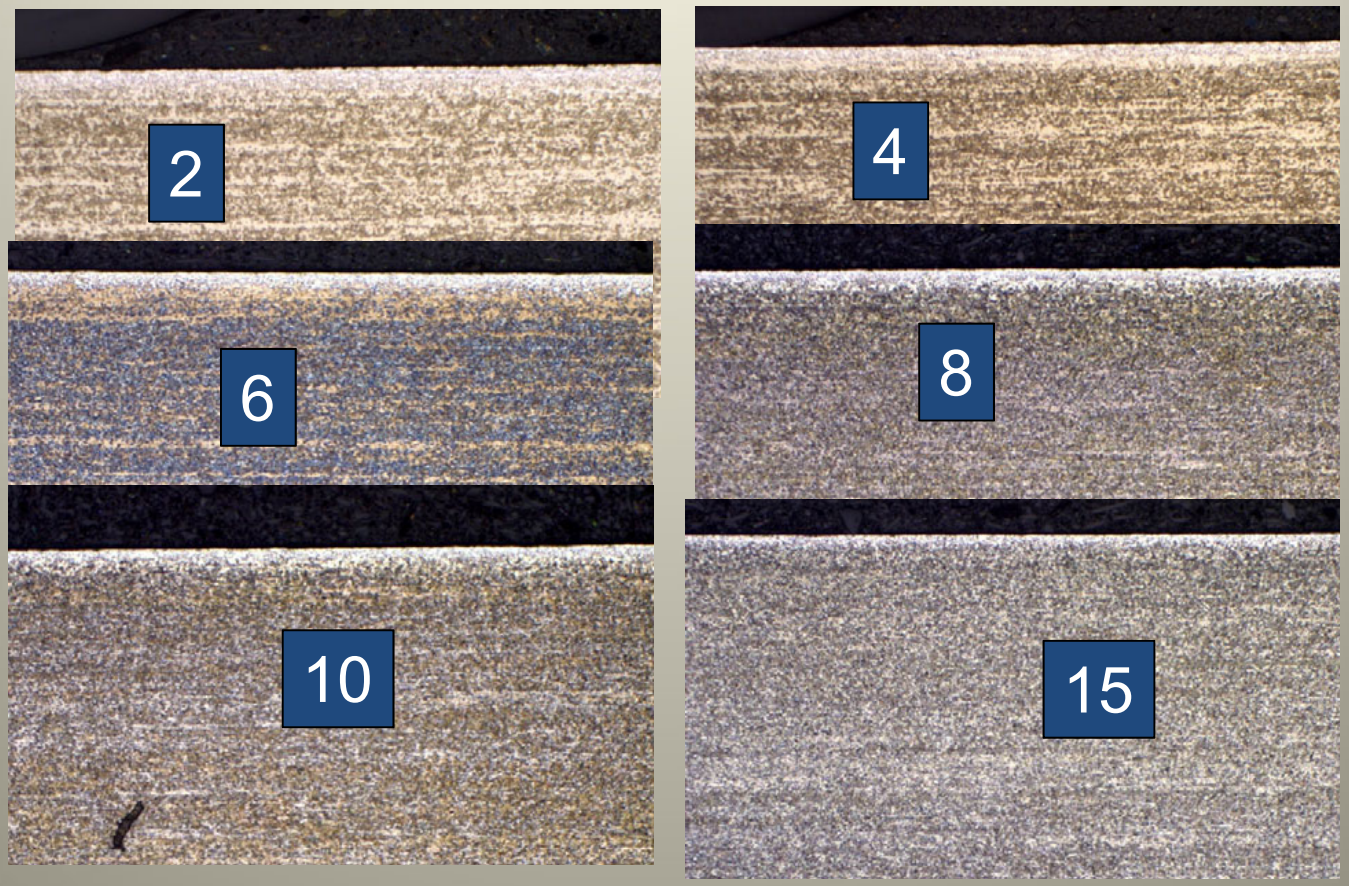






Etching Time Study

2% HF – Time in Seconds – Magnification 50X



Conclusions

- Color hues of Ti6Al4V post heat treatment is not related to the extent of alpha case that may be present
- Ramp rate did not affect the rate of alpha case formation in either the low temperature or high temperature cycles; beneficial for production
- Temperature and surface area greatly influence the extent of alpha case formation - an increase in 300°F resulted in 5 times the depth of alpha case
- RGA data reveals that water vapor is the primary oxidizing residual gas; as temperatures increase water vapor, carbon monoxide, and carbon dioxide all act as contributors (O and C) to alpha case formation
- RGA traces show the strong gettering effect of titanium for reactive gases
- Evaluation of three different metallographic etchants indicated that 2% HF is the most effective for measuring alpha case. Etchant times between 6 and 10 seconds delineated comparable alpha case depths

Titanium Heated in Air