DE-FC36-04GO14045:

Low-Temperature Surface Carburizing of Stainless Steels

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Summary of Goals and Objectives

A low temperature, i.e. 450 to 500°C, surface carburization treatment for 316 austenitic stainless steel recently was developed by the Swagelok Company that produces:

1.) A colossal supersaturation of carbon interstitials
2.) Increased surface hardness by a factor of four or five
3.) Improved corrosion and wear resistance

To realize the full potential of this technology the assembled research team will apply the process to candidate 300 and 200 series, superaustenitic and precipitation hardening stainless steels and evaluate the improvements to their properties.

The anticipated outcome will be the availability of stainless steel parts that exhibit a substantial reduction of service-induced wear in a variety of applications, e.g. impeller pumps for the chemical and petroleum industries.

By allowing pumps to operate for extended lifetimes while maintaining maximum efficiency will lead to primary energy savings on the order of $20*10^{12}$ Btu/yr. by the year 2020.

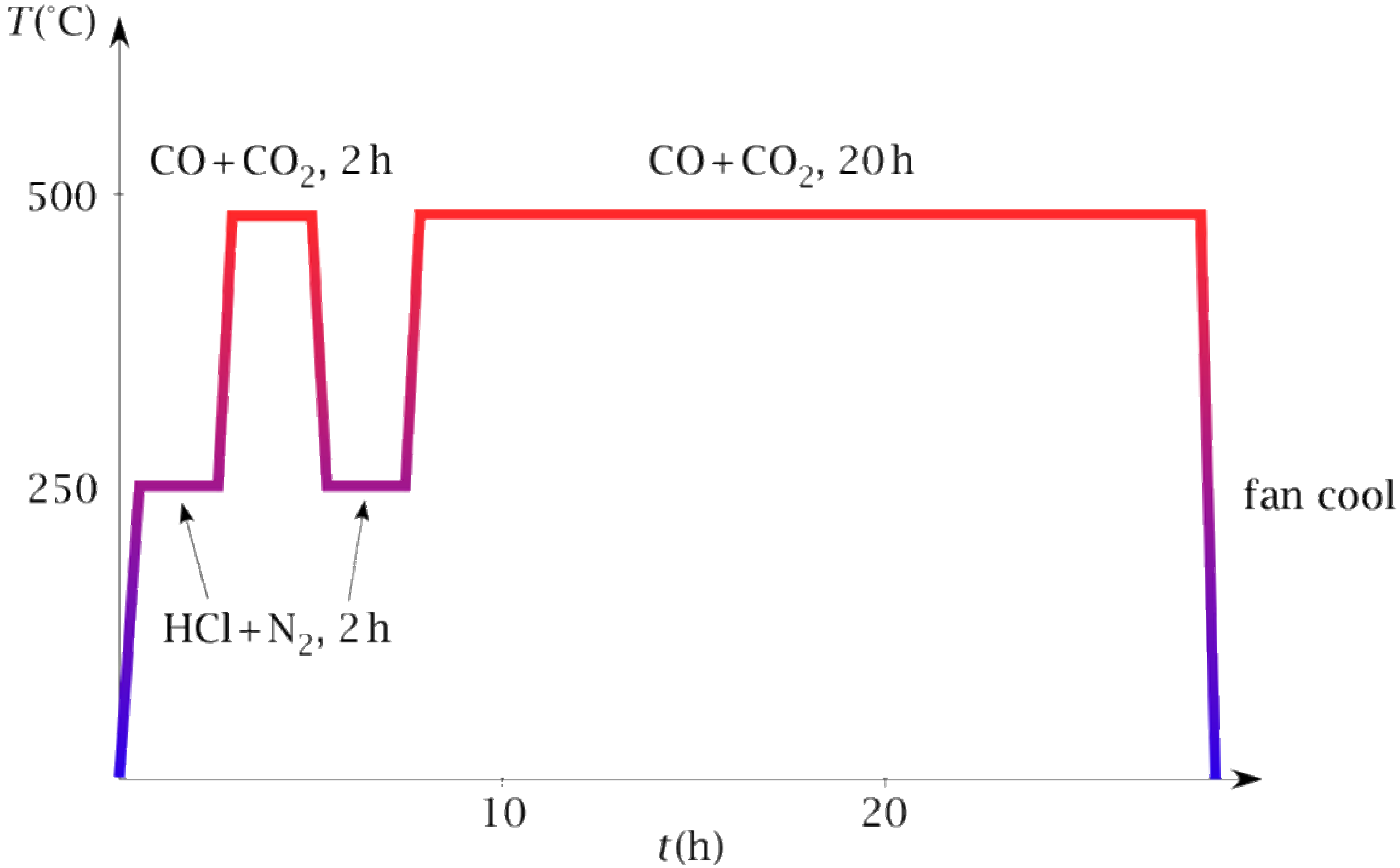
Hurdles to Be Overcome with the Candidate Alloys

- Development of appropriate surface activation procedures.
- Defining the relationships among carburization time and temperature, carbon concentration as a function of depth below the surface and the absence of carbide formation.

A Key Component of Low Temperature Carburization Is Surface Activation

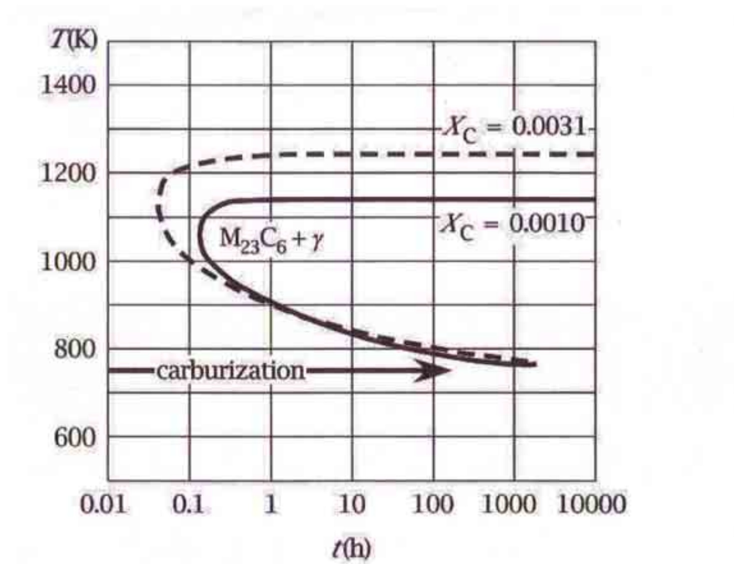
- The normal passive films on stainless steel alloys is quite resistant to carbon permeation.
- Swagelok has developed two surface treatments that remove such passive films.
  - 1.) The use of HCl gas
  - 2.) Plating a thin layer of iron on to the surface prior to carburization

A Typical Low Temperature Carburization Treatment Using HCl Surface Activation



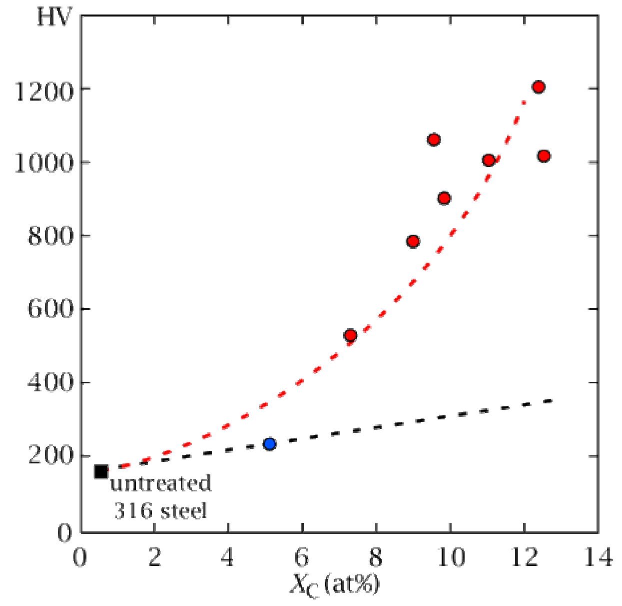
How Low Temperature Carburization Achieves Colossal Supersaturation of Interstitials

- The Time-Temperature-Transformation diagram for 316 stainless steel with two carbon levels is shown below:



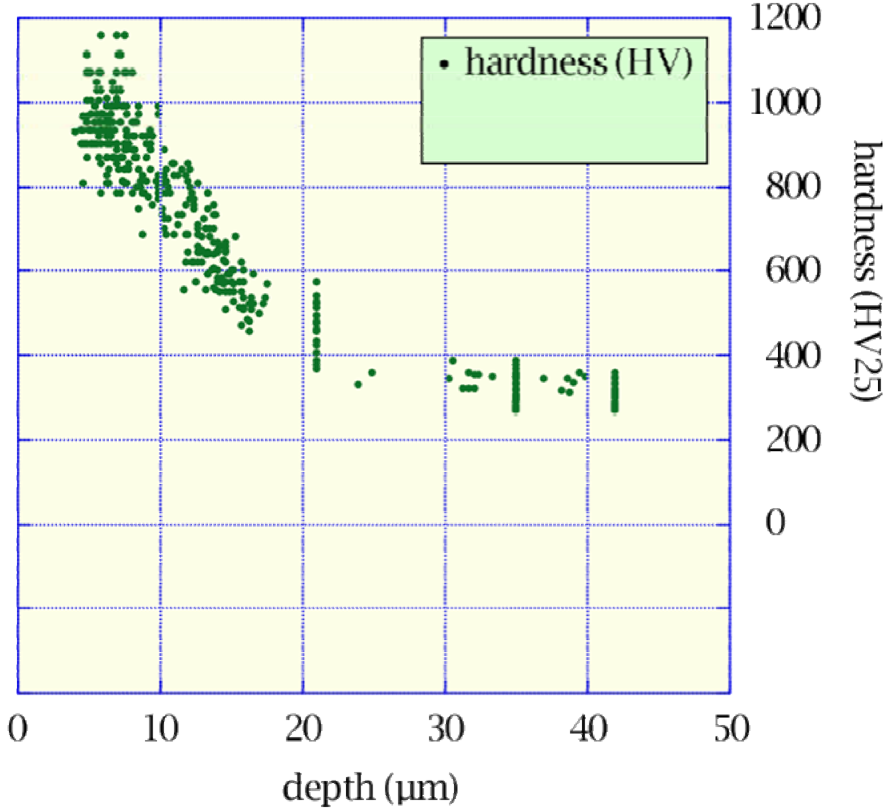
- By carburizing below the C-curve for the precipitation of $M_{23}C_6$ colossal carbon supersaturations can be attained.

Hardness As a Function of the Concentration Carbon in Solid Solution in the Austenitic Matrix of 316 Stainless Steel



Note at greater than 6 at. pct. (1.3 wt. pct.) the hardness of 316 Stainless Steel increases at a rapid rate with increasing carbon content.

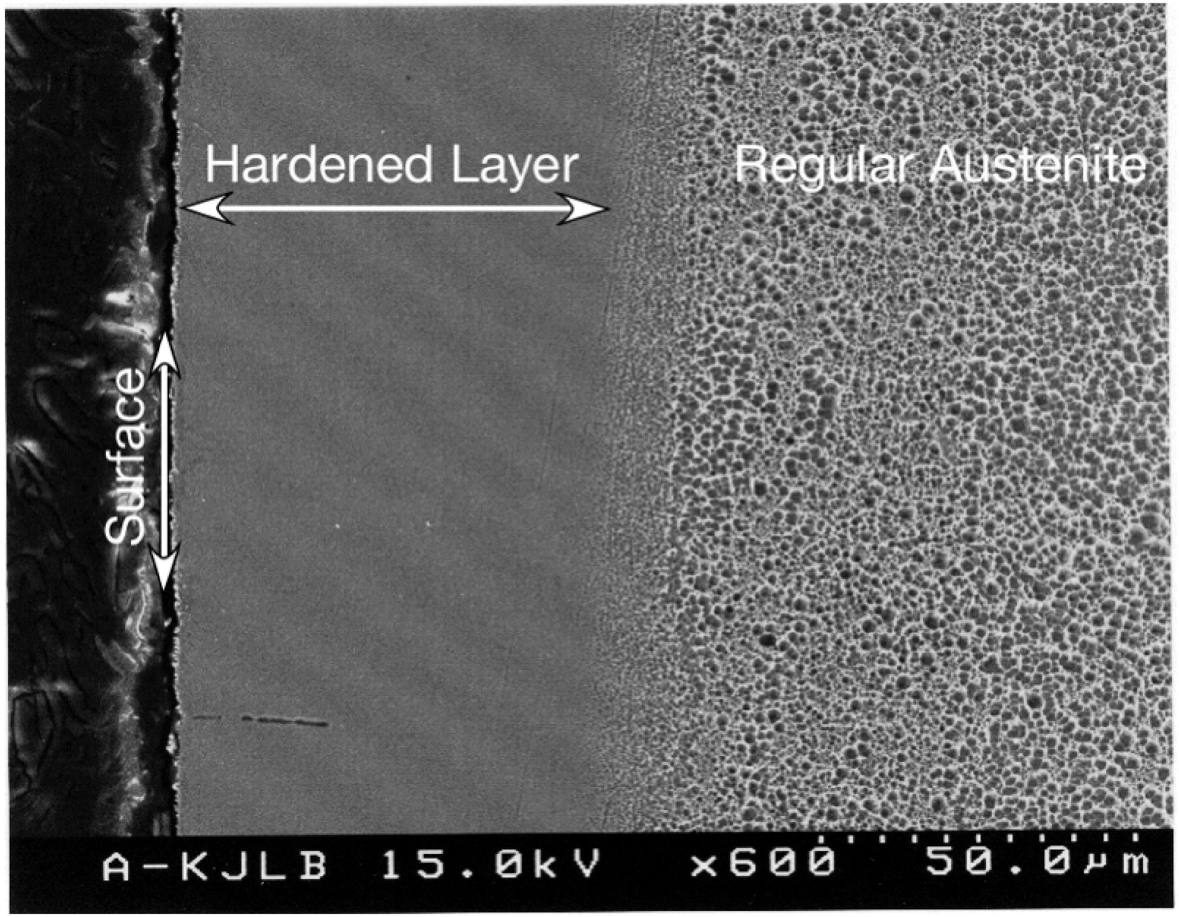
A Hardness Depth Profile from the Surface Inward for 316 Stainless Steel Subjected to a Low Temperature Carburization Treatment



Huge Compressive Surface Residual Stresses Are Generated by Low Temperature Carburization

	At.% carbon	Phase	σ_{Φ} (MPa)
Untreated 316 SS	0.2	γ	14
LTCSS treated 316 SS	10.3	Expanded γ	**–2130**

Corrosion Resistance



LTCSS treated 316 Stainless Steel etched with Marble's reagent: 10 g $CuSO_4$, 50 ml HCl, and 50 ml H_2O.

Potential Pump Components that Will Benefit from LTCSS Processing of Stainless Steels

Project Tasks

- 1) Thermodynamic Modeling of Alloy Composition
- 2) Procure Commercial and Experimental Alloys
- 3) Establish Processing Conditions Needed for Low Temperature Carburization of the Candidate Alloys
- 4) Evaluate Surface Hardness and Depth of Hardening
- 5) Characterize the Microstructures Produced
- 6) Conduct Corrosion Tests
- 7) Evaluate Tribological Properties of the Treated Alloys
- 8) Perform Fatigue Tests
- 9) Hold Technical Review Meetings and Prepare Project Reports

Anticipated Funding for the Three Year Life of the Project

	Year 1 ($ x10^3)		Year 2 ($ x10^3)		Year 3 ($ x10^3)	
Task	**DOE**	**Cost Share**	**DOE**	**Cost Share**	**DOE**	**Cost Share**
Thermodynamic Modeling	110	40	110	42.1	110	50.1
Alloy Procurement	50	10	50	10	40	10
Process Development	---	45	40	45	---	250
Hardness Measurement	---	20	30	20	---	20
Microstructured Characterization	110	45	110	47.1	110	45.1
Corrosion Testing	70	40	70	40	65	50
Tribological Evaluations	50	30	50	30	50	40
Fatigue Testing	110	10	110	10	110	10
Meetings and Reports	---	10	---	10	---	10
TOTALS:	**500**	**250**	**570**	**254.2**	**485**	**485.2**

Stainless Steels and their Compositions (by wt.%)*

Alloys:	Cr:	Ni:	Mo:	Mn:	Si:	Al:	Co:	Ti:	Nb:	V:	Cu:	W:
Inconel 625	21.5	49.95	9	0.25	0.25	0.2	…	0.2	3.65	…	…	…
203	17	5.75	0.25	5.75	0.5	…	…	…	…	…	2	…
309	23	13.5	…	1	0.5	…	…	…	…	…	…	…
317	19	13	3.5	1	0.5	…	…	…	…	…	…	…
310	25	20.5	…	1	0.75	…	…	…	…	…	…	…
AL-6XN	21	24.5	6.5	1	0.5	…	…	…	…	…	0.375	…
A286	14.75	25.5	1.25	1	0.5	0.175	…	2.125	…	0.3	…	…
304	19	9.25	…	1	0.5	…	…	…	…	…	…	…
302	18	9	…	1	0.5	…	…	…	…	…	…	…
301	17	7	…	1	0.5	…	…	…	…	…	…	…
Mp-35-N	20	35	9.75	0.075	0.075	…	34.075	0.5	…	…	…	…
316L	17	12	2.5	1	0.5	…	…	…	…	…	…	…
13-8 Mo PH	12.75	8	2.25	0.2	0.1	1.15	…	…	…	…	…	…
Incoloy 825	21.5	42	3	0.5	0.25	0.1	…	0.9	…	…	2.25	…
L-605	20	10	…	1	0.5	…	51.9	…	…	…	…	15
Nitronic 60	17	8.5	…	8	4	…	…	…	…	…	…	…

***Balance Fe**

Thermodynamic Modeling of Alloy Compositions

- Defining the solubility of carbon in austenite for the list of candidate alloys.
- Comparing the carbon solubility predictions of both CALPHAD (Thermo Calc^{TM}) and Wagner dilute solution thermodynamic models.
- Establishing the change in carbon solubility in austenite produced by potential alloying elements.

ASTM Based Mechanical Testing Anticipated

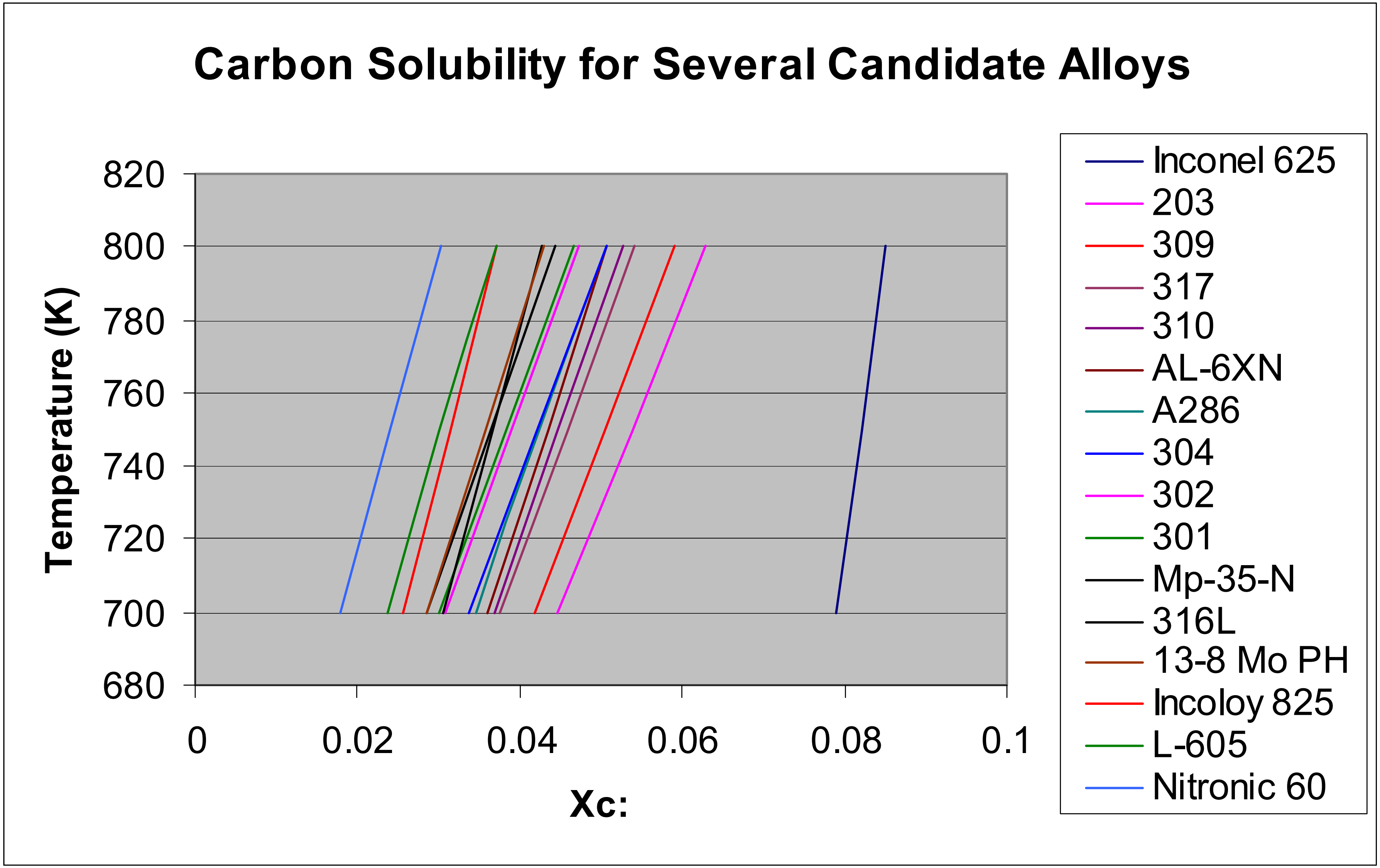
- E8 – Tension Testing
- E290 – Bend Testing
- Axial Fatigue Testing
  - E466 Stress Controlled
  - E606 Strain Controlled
- Residual Stress Measurements

Environmental ASTM Testing Proposal for Treated and Untreated Alloys

- General Corrosion Test
  - G31 Immersion Corrosion
- Electrochemical Pitting Corrosion Tests
  - G61 Cyclic Potentiodynamic Polarization
  - G150 Electrochemical Critical Pitting Temperature
- Stress Corrosion Cracking Tests
  - G129 Slow Strain Rate Environmentally Assisted Cracking

ASTM Based Tribological Evaluations

- Wear and Abrasion Tests
  - G99 Pin on Disk Wear Testing
  - G133 Reciprocating Ball on Flat (Sliding Wear)
  - G174 Low-Stress Abrasion (Loop on Flat)
- Cavitation and Erosion Tests
  - G32 Cavitation Erosion
  - G73 Liquid Impingement Erosion
  - G134 Cavitating Liquid Jet Erosion


Carbon Solubility for Several Candidate Alloys
Temperature (K)
820
800
780
760
740
720
700
680
0
0.02
0.04
0.06
0.08
0.1
Xc:
Inconel 625
203
309
317
310
AL-6XN
A286
304
302
301
Mp-35-N
316L
13-8 Mo PH
Incoloy 825
L-605
Nitronic 60

Carbon Affinities for Alloying Elements at 750 K

Cu =	72220	**Mo =**	-30400
Fe =	65290	**W =**	-36230
Ni =	46410	**Al =**	-41920
Co =	45360	**V =**	-103000
Cr =	17590	**Nb =**	-148790
Mn =	10410	**Ti =**	-155870
Si =	-26140	**Zr =**	-204850

Initial Results: Alloy Procurement

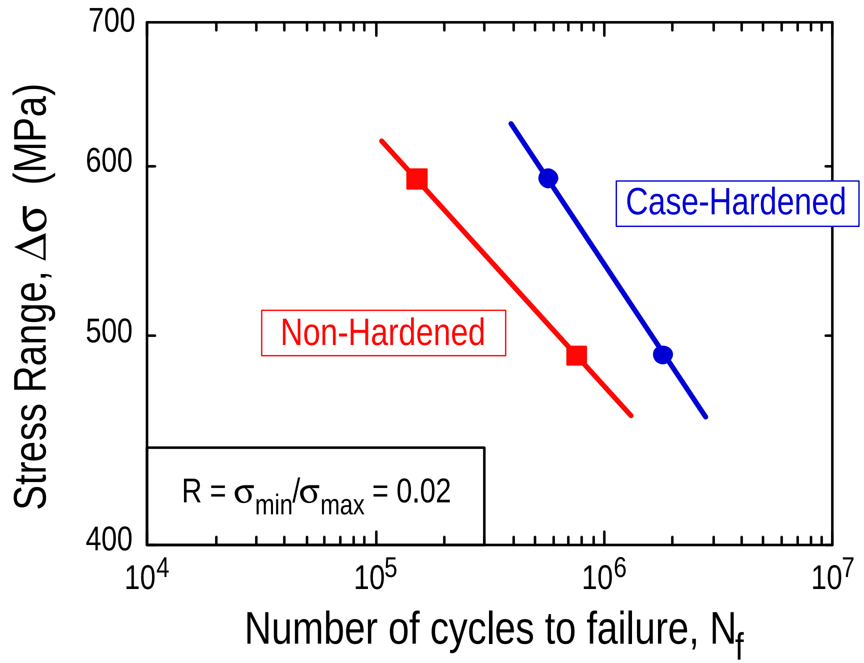
- A list of commercially available candidate alloys has been created.
- The geometries needed for each alloy based upon requirements for analysis and testing are in the process of being defined.
- Experimental alloys to be produced by arc-melting and casting 0.5x1x5 inch. ingots will be identified based upon thermodynamic modeling.

Initial Results of Corrosion Tests Run on Low Temperature Carburized 316 Stainless Steels

ASTM G150, G61

Test	Untreated	Carburized
Pitting Temperature	(16.9±0.5)°C	(79.1±3.0)°C
Pitting Potential	(343±50)mV	(945±6)mV

Initial Results: Fatigue Testing of a LTCSS Processed Alloy A286



- The A286 alloy was initially solutionized for 1 hour at 982°C, oil quenched, then aged 16 hours at 718°C producing a tensile strength of 1100 MPa.