

**ADMINISTRATIVE INFORMATION**

1. **Project Name:** Low-Temperature Surface Carburizing of Stainless Steels (CPS#16948)
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5. **Date Project Initiated:** 1 January 2004
6. **Expected Completion Date:** 31 December 2006

**PROJECT RATIONALE AND STRATEGY****7. Project Objective:**

This research program will apply a commercial patented technology, currently used for conformal surface hardening AISI 316 components, to other austenitic stainless steel components. Austenitic stainless steels that have been treated by this technology, called low-temperature colossal super-saturation (LTCSS), show increased surface hardness, improved wear resistance, improved erosion resistance, and improved corrosion resistance. The research will enable substantial reduction of the service-induced wear of

austenitic stainless steel parts in a variety of applications, including, for example, bearing surfaces and wear surfaces.

#### 8. Technical Barrier(s) Being Addressed:

Austenitic stainless steels are the primary materials of choice for applications when one needs corrosion resistance in aqueous solutions at ambient temperatures. While austenitic stainless steels are excellent for their corrosion resistance, they possess low hardness values and cannot be heat-treated to increase their hardness like the ferritic steels. Thus, when used for applications such as pumps for liquids and slurries in chemical, petrochemical, pulp and paper, steel, and other industries, they undergo wear of the pump impellers and the pump casings. The wear causes the pumps to operate with decreasing efficiency.

#### 9. Project Pathway:

Swagelok will process a group of commercially available austenitic stainless steels, and perform initial characterization, including metallography, microhardness depth profiles, carbon depth profiles, and some screening corrosion testing. CWRU will characterize the carburized specimens using materials characterization techniques such as TEM, SEM, and XRD, and will perform mechanical tests as required. CWRU will also perform thermodynamic modeling to evaluate the effects of different alloying elements on the results of the LTCSS process. ORNL will be responsible for corrosion, fatigue, and tribological testing (erosion, wear, abrasion) of a subset of the treated alloys. ORNL will also be responsible for the production of test heats of an austenitic alloy designed to optimize the results of the LTCSS process (depth of treatment and hardness of case). EIO will provide marketing support and components for treatment. Spirax Sarco will provide components for treatment and will test the components to evaluate the improvement in energy efficiency.

#### 10. Critical Technical Metrics:

Extend the LTCSS technology to other stainless steels: Thirty candidate alloys have been identified for evaluation. These alloys are readily available, commercially important in a wide range of industries, and also represent a range of compositions. Alloys will be considered successfully treated when the following parameters are achieved:

- At least double the hardness of untreated material: verified by microhardness profiles
- Uniform treatment (complete activation): verified by metallographic evaluation
- No carbide precipitation: verified by TEM and XRD analyses
- Performance improved over untreated material: verified by testing

Create pedigreed data on performance characteristics of treated alloys: When possible, standard ASTM test methods will be used, and metric rather than subjective data will be generated.

Develop understanding of effects of alloying elements on LTCSS results: By optimizing the composition of the stainless steel, much higher levels of carbon super-saturation can be reached, and these higher carbon concentrations will result in an even more impressive increase of the surface hardness and the wear- and corrosion-resistance. This concept will be examined using thermodynamic modeling.

Develop understanding of process to enable treatment of other austenitic alloys: Based on the results of the thermodynamic modeling and the wide range of compositions selected for evaluation, a model will be developed to predict the treatment of other austenitic alloys, including iron-based superalloys, nickel-based alloys, and cobalt-based alloys. The experimental heat results will be used to verify this model.

### **PROJECT PLANS AND PROGRESS**

#### 11. Past Accomplishments:

- Manufacture/procure alloys and specimens, process alloys, initial evaluations: A list of commercially available alloys was developed, and approximately 30 were down-selected for investigation. The selection was based on the alloy being austenitic, having a minimum chromium content of 12%, citation of the alloy in specifications (i.e. UNS, ASTM, SAE, etc.), and

compositional variations to explore the effects of various elements. Quantities of over 20 alloys have been obtained. Specimen geometries for the various tests have been determined, and several of the alloys have been manufactured into specimens and have undergone LTCSS treatment at Swagelok.

Microstructural characterization: Case initiated XRD analysis and TEM work on treated 316, 321, and 254 SMO specimens. Precision lattice parameters were determined for the uncarburized materials using the Nelson-Riley (N-R) fitting procedure. Significant work has gone into understanding the lattice parameter expansion of the carburized materials.

- Thermodynamic modeling: A thermodynamic model for an iron-based face-centered cubic (FCC) solid solution has been formulated using the CALPHAD two sublattices framework. The influence of Al, Co, Cr, Cu, Mn, Mo, N, Nb, Ni, Si, Ti, V, W and Zr on the solubility of C in FCC solid solutions has been assessed. The agreement between the predicted solubility under paraequilibrium conditions and the experimentally observed value at 750K is within a factor of two. As a basis for alloy development, the CALPHAD database was used to determine the change in carbon solubility under paraequilibrium conditions for additions of Cr, Ni, Si, Al, Mo, Ti, V, Nb and Zr.
- Corrosion testing: treated and untreated specimens: At Case, an experiment was performed to evaluate the effect of carbon content, microhardness, and compressive residual stress associated with carbon diffusion, on corrosion resistance. Sample disks were electropolished for 0, 40, 80, 160, 200, and 240 seconds to progressively remove the case. These specimens were then evaluated by GDOES, XRD, and XPS. Sets of the samples were also corrosion tested using ASTM G150 and ASTM G61. At ORNL, this task focuses on using an ultrasonic-based cavitation erosion testing unit for screening the potential benefits of low-temperature carburization of various alloys. Two cavitation erosion systems were ordered and the equipment has undergone validation testing. Initial tests show that untreated 316 loses weight at a rate that is approximately 5.5 times greater than the treated material.
- Mechanical Testing: Tensile testing of treated and non-treated specimens was performed at Case. In fatigue testing, carburized samples survived to higher stresses and greater numbers of cycles than the untreated samples.
- Tribological evaluations ORNL conducted initial characterization on treated and non-treated specimens of Type 316 stainless steel. This investigation included microindentation hardness tests, scratch hardness tests, and a series of pin-on-disk sliding friction and wear tests. The wear rates of couples involving treated balls and treated disk specimens were lowered by between 54 and 203 times compared to non-treated pairs
- Component testing: Components were obtained from Jeffrey Chain and from Spirax Sarco, and underwent successful treatment at Swagelok. In addition, several potential industrial partners have been identified and approached concerning alloys of interest and components for treatment.
- Design and manufacture of experimental alloy: Based on the results of the thermodynamic modeling at Case, several potential compositions were identified and provided to ORNL for manufacture.

## 12. Future Plans:

- Manufacture/procure alloys and specimens, process alloys, initial evaluations: Alloy procurement is essentially complete. Small quantities of other alloys or specimens will be obtained as needed.
- Microstructural characterization: Case will continue work on this task as other candidate alloys are successfully treated.
- Thermodynamic modeling: This task is complete and will be revisited when experimental data is available.
- Corrosion testing: treated and untreated specimens: Tests will continue at Swagelok, Case and ORNL on other candidate alloys. In addition, experiments designed to evaluate the behavior of treated and nontreated 304 and 316 in SCC and crevice corrosion will be performed.
- Mechanical Testing: Tests will continue at Case on other candidate alloys.
- Tribological evaluations: Tests will continue at ORNL on other candidate alloys.
- Component testing: Treated components will be provided to industrial partners for testing and evaluation.

- *Design and manufacture of experimental alloy:* ORNL will manufacture test quantities of the experimental alloys designed by Case. These will be treated at Swagelok, and analyzed at Case. Experimental results will be compared with predicted results from thermodynamic modeling.

13. **Project Changes:** None.

14. **Commercialization Potential, Plans, and Activities:**

Swagelok owns the original intellectual property (IP) through several issued patents (US Patents 6,093,303, 6,165,597, and 6,461,448) and others pending, and will retain ownership. Swagelok intends to follow one of two paths with regard to commercialization and technology transfer:

- Swagelok may develop a strategic business unit with the purpose of offering the LTCSS process on a toll basis to interested end users; or
- Swagelok may, on a case-by-case basis, choose to enter into exclusive licensing arrangements for the technology.

The participants of this proposal, representing industry, academia and research, will identify industries and companies whose applications have the potential of benefiting from LTCSS. Such a team of participants provides the ideal partnering possibilities for the success of implementation of technology resulting from this program in the shortest time possible. The availability of super-hard wear- and corrosion-resistant austenitic stainless steels in other applications will strengthen the US domestic industry with enhanced products. Potential applications include fasteners; bearings; medical implants (stents, joint replacements); and consumer products, such as cutlery and razors.

15. **Patents, Publications, Presentations:**

A poster presentation describing the project was made in Arlington, VA on June 24, 2004 at the IMF Portfolio Review.

A paper entitled "Surface Hardening of Austenitic Steels by Low Temperature Colossal Supersaturation" was submitted for publication in the MS&T'04 Volume II – Engineering Steel Surfaces: AIST/TMS Proceedings. A presentation entitled "Surface Hardening of Austenitic Steels by Low Temperature Colossal Supersaturation" was delivered at 8:30 a.m. on Wednesday, September 29, 2004 in the Symposium on Engineered Steel Surfaces: Nanotechnology, Hardness and Wear Resistance at MS&T'04 in New Orleans. In both cases the authors are G. M. Michal, F. Ernst and Y. Cao.

A paper entitled "Wear Reduction of Austenitic Stainless Steels using a Novel, Low-Temperature Carburization Process," has been submitted and accepted for the World Tribology Congress III to be held in Washington, DC in September 2005. The authors are P. J. Blau, J. Qu, and B. C. Jolly. It describes the results of sliding friction and wear studies that have been completed to date under the auspices of this project.

A manuscript entitled "Impressive Carbon Supersaturation due to Paraequilibrium Carburization of Austenitic Stainless Steel," has been written and is under review for publication in *Science*. The authors are G.M. Michal, F. Ernst, H. Kahn, Y. Cao, F. Oba, and A. H. Heuer.

A paper entitled "Surface Engineering of Stainless Steel to Improve Wear Resistance without Sacrificing Corrosion Resistance" has been submitted for presentation at the 19th International Conference on Surface Modification Technologies (SMT 19) to be held on Aug. 1-3, 2005 in St. Paul, Minnesota. The title is The authors are Peter J. Blau, Jun Qu, and B. C. Jolly.