Wear 265 (2008) 1909–1913

Contents lists available at ScienceDirect

Wear

journal homepage: www.elsevier.com/locate/wear

Short communication

Effects of multiple treatments of low-temperature colossal supersaturation on tribological characteristics of austenitic stainless steels

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ARTICLE INFO

Article history:
Received 4 December 2007
Received in revised form 25 February 2008
Accepted 26 March 2008
Available online 8 May 2008

Keywords:
Stainless steel
Low-temperature carburization
Multiple treatment
Wear-resistance

ABSTRACT

An alternative carburization process, low-temperature colossal supersaturation (LTCSS), has demonstrated significant improvement on both wear- and corrosion-resistance for austenitic stainless steel surfaces in recent literature. This study explores the effects of multiple treatments of LTCSS on tribological characteristics for Type 316 stainless steel. Thicker carburized layers were produced by multiple LTCSS treatments, with 30, 45, and 55 μm for one, two, and four treatments, respectively. Although the hardness remains unchanged at low-load microindentation, multiple treatments have showed higher values in both microindentation and scratch hardness tests when deeper penetrations occurred under heavier loads. The friction and wear characteristics of Type 316 stainless steel with multiple LTCSS treatments were evaluated in non-lubricated unidirectional sliding (pin-on-disk) against Type 440C stainless steel. While little change was observed on friction behavior, substantial further improvement on wear-resistance has been achieved for the multiple treatments. In addition, the wear of the counterface was also largely reduced when rubbing against a multiply treated surface.

1. Introduction

Carburization has been employed for at least 2000 years [1–3] to harden the surfaces of iron-based alloys. The wear resistance of stainless steels can be enhanced by traditional carburization, however their corrosion resistance is usually reduced due to the formation of chrome carbides that decrease the ability to form a protective chromium oxide film on the surface. A new carburization process, so called low-temperature colossal supersaturation, for austenitic stainless steels was recently developed to improve the surface hardness and wear-resistance without sacrificing the corrosion resistance [4–7]. Low-temperature colossal supersaturation (LTCSS) enables up to 12 at.% carbon to be diffused into the stainless steel surface without causing precipitation of chrome carbides. A surface activation step is used to remove the protective chromia (Cr_2O_3) film by dry HCl gas at 250 °C, which enables more effective carbon diffusion. The post-activation carburization treatment is done at 470 °C for 20–30 h. At this temperature (470 °C), 'paraequilibrium', rather than conventional thermodynamic equilibrium, determines the phase composition to allow for vastly increased solubility limits for carbon. Detailed mechanisms and kinetics including a phase diagram and carbon solubility in paraequilibrium are discussed in [6]. Our previous work reported significantly improved wear-resistance by a single-treatment of LTCSS for Type 316 stainless steels [8]. This study, as a follow-on work, investigates the tribological characteristics of Type 316 stainless steel that received multiple treatments of LTCSS.

2. Materials and experimental procedure

Eight Type 316 stainless steel disk specimens of 25 mm in diameter and 6.35 mm in thickness were made. Six disks were treated by LTCSS with two at each condition of single treatment (T-1X), double treatments (T-2X), and four treatments (T-4X) at Swagelok Company, Ohio, USA. The optical micrographs of the etched (by 4% Nital etchant) cross-sections are shown in Fig. 1. The thickness of the carburized layer is about 30, 45, and 55 μm for T-1X, T-2X, and T-4X, respectively. Unlike the Type 316 stainless steel substrate whose microstructures were clearly revealed by etching, the carburized layers were resistant to the acid etchant. This supports their improved corrosion resistance reported by previous studies [4–6].

Disk surfaces were metallographically polished (R_a: 0.016 μm) before treatments. The surfaces were slightly rougher after each treatment, as shown in Table 1. The carburized disks were tested as received.

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doi:10.1016/j.wear.2008.03.011

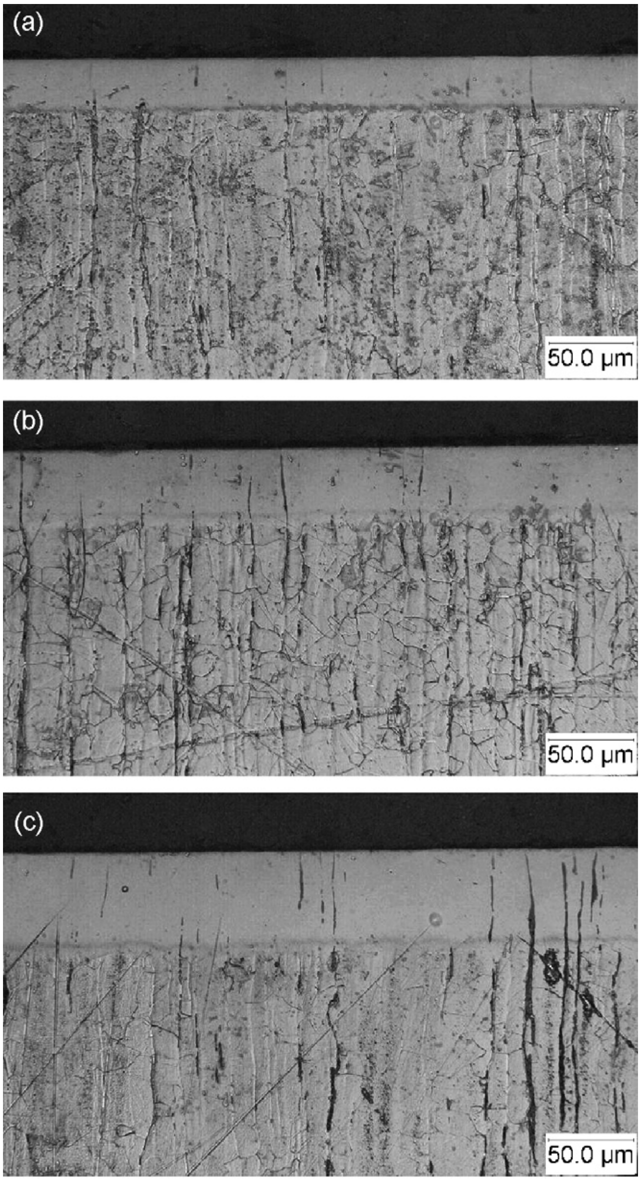

Fig. 1. Etched cross-sections of Type 316 stainless steel surfaces with multiple LTCSS treatments. (a) T-1X; (b) T-2X; (c) T-4X.

Vickers microindentation hardness (Buehler Micromet® 2100™) and scratch hardness (CSM Revetest®, using a spherically tipped diamond stylus with a 200-μm tip radius) tests were conducted on both treated and non-treated Type 316 stainless steel surfaces.

Friction and wear characteristics of the carburized and non-treated Type 316 stainless steel were evaluated using a unidirectional sliding ball-on-disk apparatus [9] at room temperature without intentional lubricant (dry). The counterface (slider) was a Type 440C stainless steel ball (Grade 25) with a diameter of 9.525 mm. The Vickers microindentation hardness of the ball slider is 9.56 and 9.11 GPa at 0.245 and 4.905 N loads, respectively. The same testing parameters were used in all tests: a load of 5 N, a sliding speed of 0.1 m/s with a wear track diameter of 10 mm, and a total sliding distance of 500 m. At least two replicates were conducted at each testing condition. The friction force was measured in situ by a load cell and used to calculate the friction coefficient. The wear volumes were determined by wear scar/track profiling using a new single-trace method described previously [10]. Wear rates were calculated by dividing the volumetric material loss by the normal load and sliding distance.

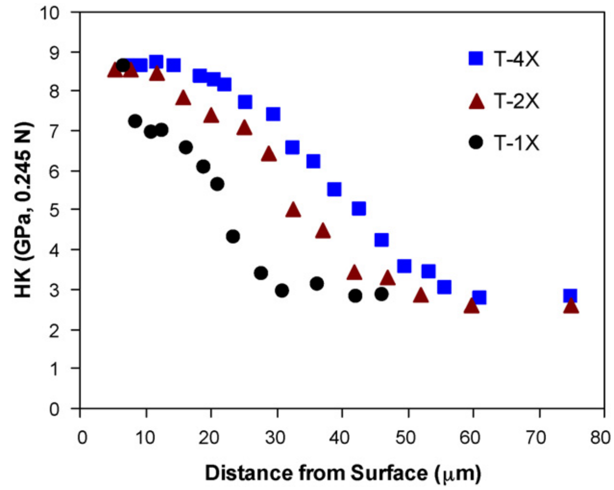

Fig. 2. Hardness profiles on the cross-sections of Type 316 stainless steel surfaces with multiple LTCSS treatments.

3. Results and discussion

3.1. Hardness

The microindentation and scratch hardness results are summarized in Table 1. The Vickers microindentation at a low load of 0.245 N produced similar hardness (10–11 GPa) for all treated surfaces, independent of the number of treatments. As contrast, the Vickers microindentation hardness at a higher load of 4.905 N and the scratch hardness at a load of 9.81 N clearly distinguished T-1X, T-2X, and T-4X. This makes sense, because multiple LTCSS does not further increase the carbon concentration compared to single treatment that already creates carbon super-saturation but does increase the carburization depth. Hardness transition profiles through the treated layers were revealed by Knoop microindentation (0.245 N) on the cross-sections, as shown in Fig. 2.

3.2. Friction

The friction behavior over the test period was different for the non-treated and treated surfaces. As shown in Fig. 3(a), the friction

Table 1
Characterization of non-treated and multiple LTCSS treated Type 316 stainless steel

	Treated layer thickness (μm)	Surface roughness R_a (μm)	Microindentation Vickers hardness (HV, GPa)		Scratch hardness, HSp (GPa, 9.81 N)
			0.245 N	4.905 N	
NT	–	0.016	2.19 ± 0.03	1.70 ± 0.04	2.45 ± 0.07
T-1X	30	0.074	10.23 ± 0.09	5.53 ± 0.19	5.55 ± 0.09
T-2X	45	0.079	10.60 ± 0.16	8.67 ± 0.52	8.36 ± 0.20
T-4X	55	0.094	10.94 ± 0.44	9.19 ± 0.25	10.21 ± 0.13

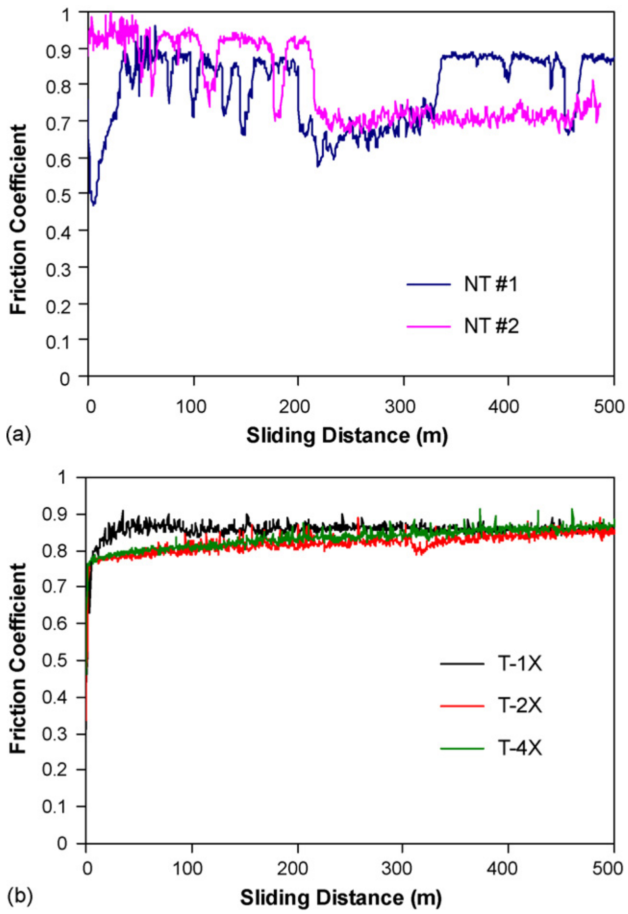

Fig. 3. Friction behavior during sliding tests against Type 440C stainless steel. (a) Non-treated Type 316 stainless steel disks; (b) LTCSS-treated Type 316 stainless steel disks.

coefficient in both tests of the non-treated surface showed unstable behavior, fluctuating between two levels: a higher level 0.85–0.90 and a lower level 0.65–0.70—implying that a transfer layer builds up and collapses during sliding. The first test (NT #1) finished at the higher friction level and the second test finished at the lower friction level (NT #2) at the end of the 500 m sliding. The wear scar morphology of the Type 440C stainless steel ball sliders after these two tests are quite different, as shown in Fig. 4. The worn spot produced in NT #2 is fully covered by a thick transfer layer, but the wear scar of NT #1 has only minimum amount of transfer material. The transfer layer seems to be a mixture of relatively large-sized reflective chips and fine reddish particles. The reflective chips are probably of metallic phase (mostly from the soft non-treated Type 316 stainless steel disk) and the reddish particles are probably mainly composed of iron oxides (Fe_2O_3). The hardness of Fe_2O_3 formed in wear testing is not readily available, but may be estimated by the value of thermally oxidized Fe_2O_3 scales, which was reported to be 6.7 GPa HV [11]. Therefore, the transfer layer is softer compared to the slider material Type 440C stainless steel, and probably reduces friction coefficient. This helps explain the friction fluctuations during testing: the metal–metal contact produces a higher level friction coefficient (after running-in), the wear debris accumulates to build up a transfer layer that temporarily reduces the friction coefficient to a lower level, and the transfer layer becomes thicker/unstable and eventually spalls off to allow metal–metal contact again when the friction coefficient is back to the higher level.

The friction behavior of the LTCSS-treated surfaces was less complex, basically a typical short running-in period followed by a steady-state stage, as shown in Fig. 3(b). Limited amount of transferred material was observed on the ball wear scars, but not in any significant coverage. The steady-state values were in a range of 0.80–0.85 and fall between the two levels of friction coefficient produced by the non-treated surfaces. There is no apparent difference among T-1X, T-2X, and T-4X.

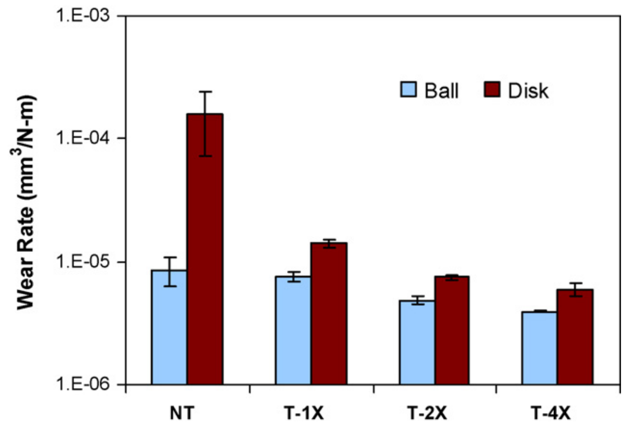

Fig. 5. Wear rates for non-treated and LTCSS-treated Type 316 stainless steel against Type 440C stainless steel.

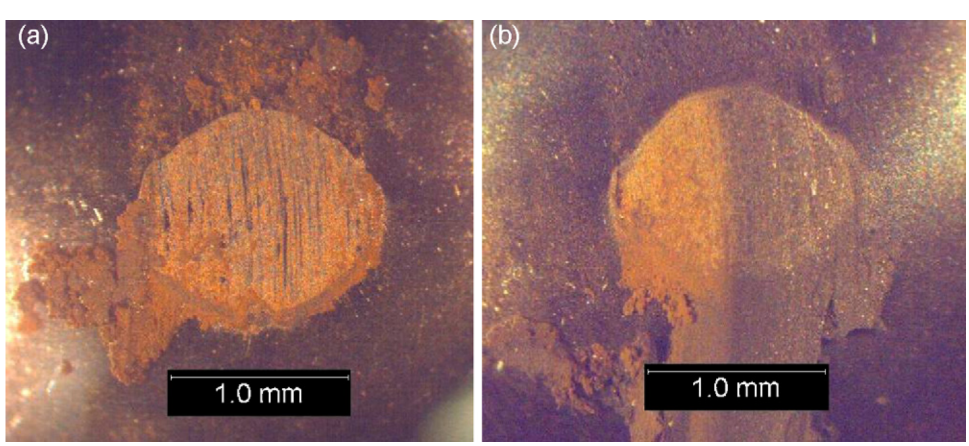

Fig. 4. Wear scars on ball sliders against non-treated Type 316 stainless steel disks. (a) NT #1; (b) NT #2.

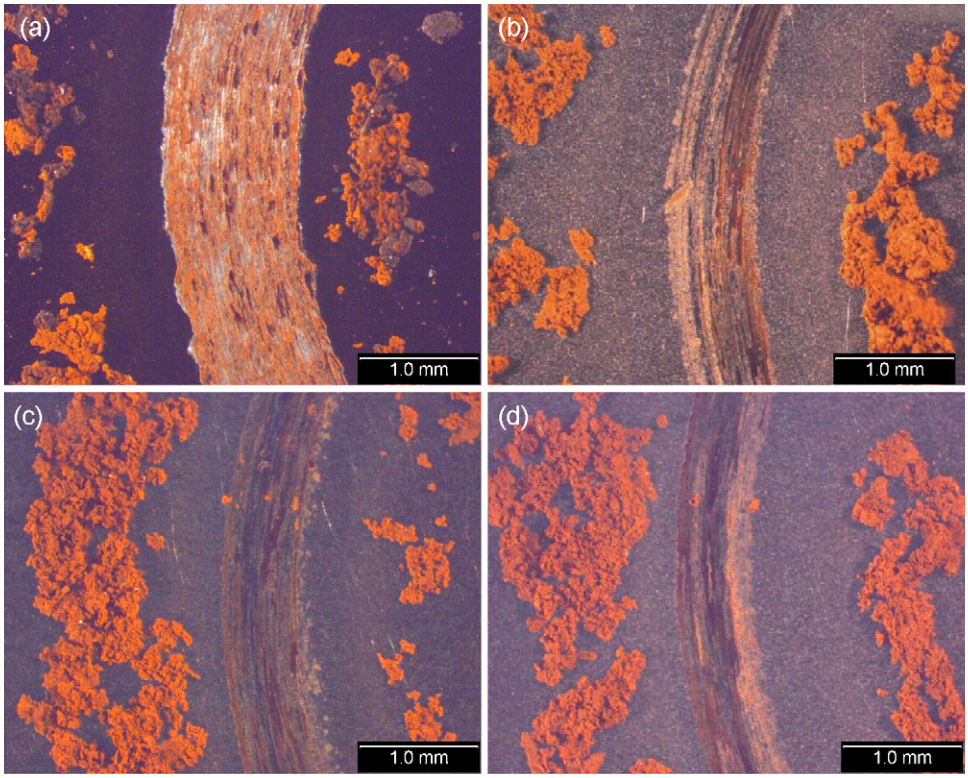

Fig. 6. Wear tracks on non-treated and LTCSS-treated stainless steel disks. (a) NT; (b) T-1X; (c) T-2X; (d) T-4X.

3.3. Wear

The wear rates of the non-treated and LTCSS-treated disks and their counterface (sliders) are summarized in Fig. 5. The relatively small error bars indicate good test repeatability. Results clearly show that LTCSS significantly reduced the wear rate for Type 316 stainless steel. The first treatment produced the biggest improvement by one order of magnitude and the multiple treatments further improved the wear-resistance but had less significant effect. The T-4X surface had a wear rate about 26 times lower than the NT surface. The wear tracks on the non-treated and treated disks are compared in Fig. 6. Apparently, the scar width was narrower on a disk with more LTCSS treatments. Unlike the wear debris produced by the non-treated disks that is a mixture of fine iron oxide particles and large metallic flakes, the wear debris generated by treated disks is dominated by those reddish iron oxides.

It was noticed that the wear rate of the counterface (Type 440C stainless steel balls) was also reduced when sliding against the treated disks, by 11, 43, and 54% for T-1X, T-2X, and T-4X, respectively. This indicates that the LTCSS-treated surfaces, though harder and more wear-resistant, are actually less abrasive to the counterface than the non-treated ones.

Note that the wear rate reported here only represents the average rate of material loss in the testing period. One important factor of the wear process is the contact pressure, which usually nonlinearly changes during the sliding test [12]. The moduli of elasticity of the NT and T-1X Type 316 steel surfaces were determined to be 179 and 200 GPa, respectively, using nanoindentation [8]. Since the carbon content is already 'supersaturated' in the T-1X surface, multiple treatments are not expected to significantly further change the mechanical properties in the near surface zone. Therefore, the moduli of elasticity of T-2X and T-4X surfaces can be assumed to be 200 GPa. The modulus of elasticity of the Type 440C stainless steel is 200 GPa [13]. Assuming the Poisson's ratios of the stainless steels tested in this study are 0.3, the mean Hertzian contact stress at the beginning of a sliding test can be calculated: 515 MPa for the NT surface and 535 MPa for the treated surfaces. By measuring the sizes of the wear scars on the balls, the mean contact pressures at the end of the sliding tests were estimated to be in the range of 4–6 MPa for both non-treated and treated surfaces, two orders of magnitude lower than the initial contact pressures. Therefore, one would expect a higher wear rate initially and a lower wear rate at the end of the test.

4. Conclusion

Tribological properties of Type 316 stainless steel with multiple treatments of LTCSS were investigated. The following conclusions have been drawn:

(1) The microindentation hardness and scratch hardness of the treated Type 316 stainless steel surfaces were up to five times greater than those of the non-treated surfaces. Multiple treatments produced thicker carburized layers that showed higher surface hardness and scratch-resistance under heavier loads.
(2) Under dry sliding against Type 440C stainless steel at room temperature, the treated surface provided more stable friction behavior by substantially reducing the adhesive wear and material transfer. The T-1X surface demonstrated wear reductions by 11 times for itself and 11% for its counterface, compared with the NT surface. Multiple treatments further enhanced the wear-resistance, such as T-2X and T-4X had wear rates 21 and 26 times less than NT, respectively. At the mean time, the counterface also wore less (up to 54%) against the multiple-treated surfaces.

Acknowledgments

The authors gratefully acknowledge the assistance of Peter Williams of Swagelok Co. for providing the treatments on test specimens. This effort was funded by the U.S. Department of Energy, Office of Advanced Industrial Materials, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

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