

Hardness of Carburized Surfaces in 316LN Stainless Steel after Low Temperature Neutron Irradiation

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Metals and Ceramics Division

**HARDNESS OF CARBURIZED SURFACES IN 316LN STAINLESS STEEL
AFTER LOW TEMPERATURE NEUTRON IRRADIATION**

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ABSTRACT

A proprietary surface carburization treatment is being considered to minimize possible cavitation pitting of the inner surfaces of the stainless steel target vessel of the SNS. The treatment gives a large supersaturation of carbon in the surface layers and causes substantial hardening of the surface. To answer the question of whether such a hardened layer will remain hard and stable during neutron irradiation, specimens of the candidate materials were irradiated in the High Flux Isotope Reactor (HFIR) to an atomic displacement level of 1 dpa. Considerable radiation hardening occurred in annealed 316LN stainless steel and 20% cold rolled 316LN stainless steel, and lesser radiation hardening in Kolsterised layers on these materials. These observations coupled with optical microscopy examinations indicate that the carbon-supersaturated layers did not suffer radiation-induced decomposition and softening.

1. INTRODUCTION

In the Spallation Neutron Source (SNS), a 316LN austenitic stainless steel vessel will be used to hold the flowing liquid mercury target [1,2]. Although 316LN steel is known to have excellent resistance to general corrosion and to retain excellent ductility after irradiation, the interior surface of the vessel could be subjected to pitting erosion due to collapse of cavities created in the mercury by the pulsed proton beam [1]. This type of cavitation pitting is common in hydraulic systems where perturbed flow is encountered [3,4], and it generally can be diminished by increasing the hardness (strength) of the vessel [5]. Several techniques have been considered to mitigate concerns about pitting erosion in the SNS target vessel. One technique that has shown good promise is a commercial surface hardening treatment known as Kolsterising[®] [3].

Kolsterising[®] is the registered trade name of a proprietary surface carburization treatment for austenitic alloys provided by Bodycote Kolsterising[®] North America, Boaz, Alabama. Kolsterising[®] is a process in which carbon is diffused into the surface of an austenitic alloy at low temperature. Bodycote's regular treatment purportedly affects a layer about 33 μm deep. Within the layer, the infiltrated carbon is claimed to be incorporated in supersaturated interstitial solid solution in the austenite phase of the steel. Special advantages are said to be improved resistance to pitting, stress corrosion, and crevice corrosion; increased resistance to wear and galling; and greater fatigue properties [3].

To verify these promotional claims and to uncover any factors that might be of concern for the integrity of a Kolsterised target vessel, the surface layers of Kolsterised austenitic 316LN stainless steel have been characterized at ORNL [4,5]. The carburized layer was about 35 μm thick with some variations. Its indentation hardness (by 50-g load) at the surface was about 1040 $\text{HV}_{0.05}$, five times larger than that of the substrate steel, $\sim 200 \text{HV}_{0.05}$, and declined rapidly with depth into the layer following the carbon concentration gradient. The corrosion resistance of the carburized layer in an acid medium was greater than that for untreated austenite. Even with the high hardness, the layer was plastically deformable and was quite resistant to cracking during straining. The maximum carbon content of the layer was much less than the claimed 6-7 wt% carbon, and the carbon was not simply contained in supersaturated solid solution; some of it was present in a previously unreported iron carbide phase located at the very surface. Inclusion stringers and ferrite phase embraced in the treated layer were less resistant to chemical attack than the treated austenite.

Although the above findings support most of the advertised claims for the Kolsterized layer, those did not include radiation effects. Irradiation usually hardens a material; however, it can soften a material that has previously been hardened by other mechanisms. In particular, irradiation can cause decomposition of metastable solid solutions [6,7]. Such decomposition might reduce the hardness and pitting resistance of a Kolsterised surface layer. To determine whether radiation-induced softening is likely to occur in the SNS target vessel during operation, the hardness of a Kolsterised layer has been measured after neutron irradiation. The results are reported herein.

2. EXPERIMENTAL

Disk samples intended for transmission electron microscopy (TEM) studies, and slivers cut from TEM disks in a variety of material conditions, were irradiated in the Hydraulic Tube facility of the HFIR to a neutron fluence of $1.2 \times 10^{25} \text{ n.m}^{-2}$ ($E > 0.1\text{MeV}$), corresponding to an atomic displacement dose of ~ 1 dpa, at a temperature of 60–100°C in direct contact with flowing water coolant. The 316LN stainless steel specimens were irradiated in 6 different conditions: as-annealed, annealed-and-Kolsterised, 20% (or 74%) cold-rolled (CR), and 20% (or 74%) CR-and-Kolsterised conditions. The Kolsterising[®] treatments were conducted by Bodycote to a 33 μm depth. Of these materials and conditions, the annealed and 20% CR 316LN stainless steels are the most probable substrate choices for the SNS vessel. To date, only limited on-face microhardness testing and visual examination of the Kolsterised and non-Kolsterised surfaces have been performed on those selected materials.

Hardness tests were performed in an automatic micro-Vickers hardness testing system (Mitsutoyo AAV-500). Because the Kolsterised layer is only about 35 μm thick, microhardness indentations made at a high load will penetrate through the hardened layer and give falsely low readings. Bodycote Group uses a load of 50 g. We found that at 50 g the diagonal lengths of the pyramidal impressions in Kolsterised surfaces were about 10 μm , which is less than one third of the thickness of Kolsterised layer. Therefore, a hardness value measured at 50-g load is believed to give a good representation of the strength of the hardened layers. Five or six hardness measurements were made for each specimen, and the dwell time at a constant load in each indentation test was 10 seconds. Optical microstructures were observed using the microscope of the hardness testing system, and the images were saved to electronic files.

3. RESULTS AND DISCUSSION

The mean values of the on-face hardness values are presented in Fig. 1, along with the statistical bounds ($\pm 1\sigma$), and all measurements are listed in Tables 1 and 2. The annealed material without Kolsterising[®] treatment showed the biggest increase in hardness by irradiation, an 81% increase from 162 HV_{0.05} before irradiation. The hardness of the Kolsterised layer on the annealed material layer was about 939 HV_{0.05} before irradiation, which is about 5.8 times higher than that of the as-annealed surface, and was increased by 11% to 1043 HV_{0.05} after irradiation.

Because of the cold work in the 20% CR material, the hardness before irradiation was about 80% higher than the annealed material, and it increased about 43% to 418 HV_{0.05} after irradiation. Kolsterising[®] raised the surface hardness to 983 HV_{0.05}, not quite as hard as the layer on the annealed substrate. This small difference may be due to the expected presence of small quantities of strain-induced martensite phase in the 20%CR material. According to Bodycote, non-austenitic phases are less responsive to Kolsterising[®] than is austenite. Irradiation increased the hardness value for the Kolsterised layer in the 20%CR material very slightly to 1003 HV_{0.05}, which is within the experimental error bounds for the Kolsterised 20%CR material before irradiation.

These hardness data indicate no signs of radiation-induced softening in the Kolsterised layers on both the annealed and 20%CR substrate materials. On the contrary, irradiation actually increased the hardness of the annealed + Kolsterised condition.

The integrity of the Kolsterized layers after irradiation is also tentatively confirmed through microstructural observations. Figures 2 and 3 present typical microstructures and indentation impressions on the tested surfaces. Some rusting or surface contamination that occurred during irradiation or post-irradiation storage, Figs. 2(b), 3(b), and 3(d) hampered the observations. Nevertheless, careful examinations revealed no convincing evidence of decomposition of the irradiated Kolsterised layers. The diagonal width of an indentation impression decreased from about 25 μm in the as-annealed condition to about 9.5 μm in the Kolsterised conditions. Slip bands were developed around the indentations. No cracking or flaking at impressions was observed, even on irradiated Kolsterised surfaces. In short, the Kolsterised layers do not seem to have been adversely affected by the neutron irradiation.

In the absence of detailed TEM studies of the microstructures of the irradiated Kolsterised layers, the present findings can not be taken as proof that the Kolsterised layers were fully resistant to irradiation-induced decomposition. But they do indicate that if some decomposition did occur it must have been on such a fine scale that it was not resolvable in the optical microscopy examinations and was not coarse enough to affect the hardness measurements, which would mean that the decomposition products would be no larger than about 1 μm . It is possible that radiation hardening of the Kolsterised layers might have offset the softening effects of any fine decomposition, resulting in a small net effect in microhardness.

4. CONCLUSIONS

Neutron irradiation of SNS target vessel materials to an atomic displacement level of 1 dpa produced considerable radiation hardening in annealed 316LN stainless steel and 20% cold rolled 316LN stainless steel, and relatively minor hardening of Kolsterised (carburized) layers on these materials. These observations coupled with optical microscopy examinations indicate that the carbon-supersaturated surface layers did not suffer significant radiation-induced decomposition and softening.

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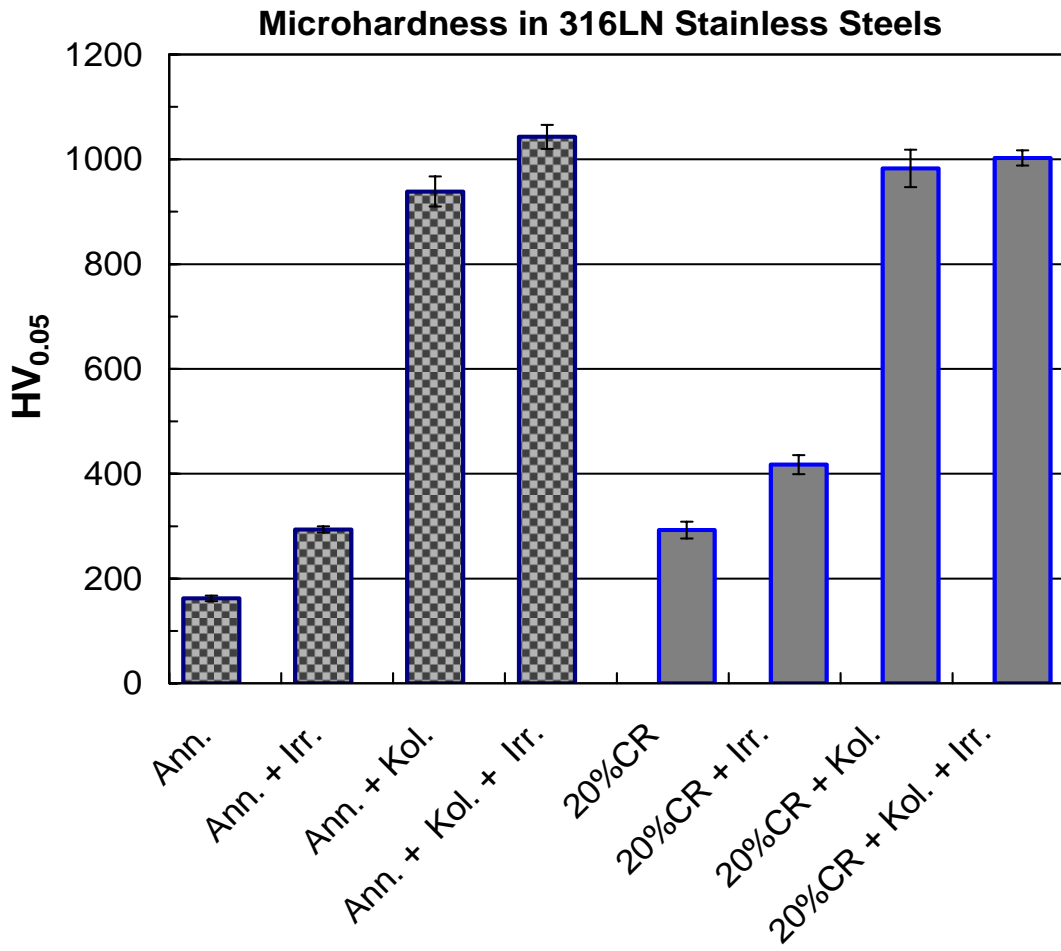


Fig. 1. Comparison of microhardness data for different material and surface conditions.

Table 1. Microhardness data for annealed 316LN stainless steel disks

Material condition	HV_{0.05}	Mean	Standard deviation (σ)	ΔHV (Irradiation hardening)
As-annealed	167	162	5	
	164			
	162			
	165			
	162			
Annealed + irradiated	153	294	6	132
	288			
	298			
	299			
	286			
Annealed + kolsterised	298	939	29	
	965			
	900			
	946			
	918			
Annealed + kolsterised + irradiated	927	1043	23	104
	975			
	1017			
	1072			
	1027			
	1061			
	1038			

Table 2. Microhardness data for 20% cold rolled 316LN stainless steel disks

Material condition	HV_{0.05}	Mean	Standard deviation (σ)	ΔHV (Irradiation hardening)
20% CR	281	293	16	
	312			
	310			
	280			
	298			
	275			
20% CR + irradiated	401	418	18	125
	438			
	429			
	396			
	423			
	918			
20% CR + kolsterised	985	983	36	
	985			
	1027			
	996			
	985			
	995			
20% CR + kolsterised + irradiated	1006	1003	14	20
	1006			
	1027			
	996			

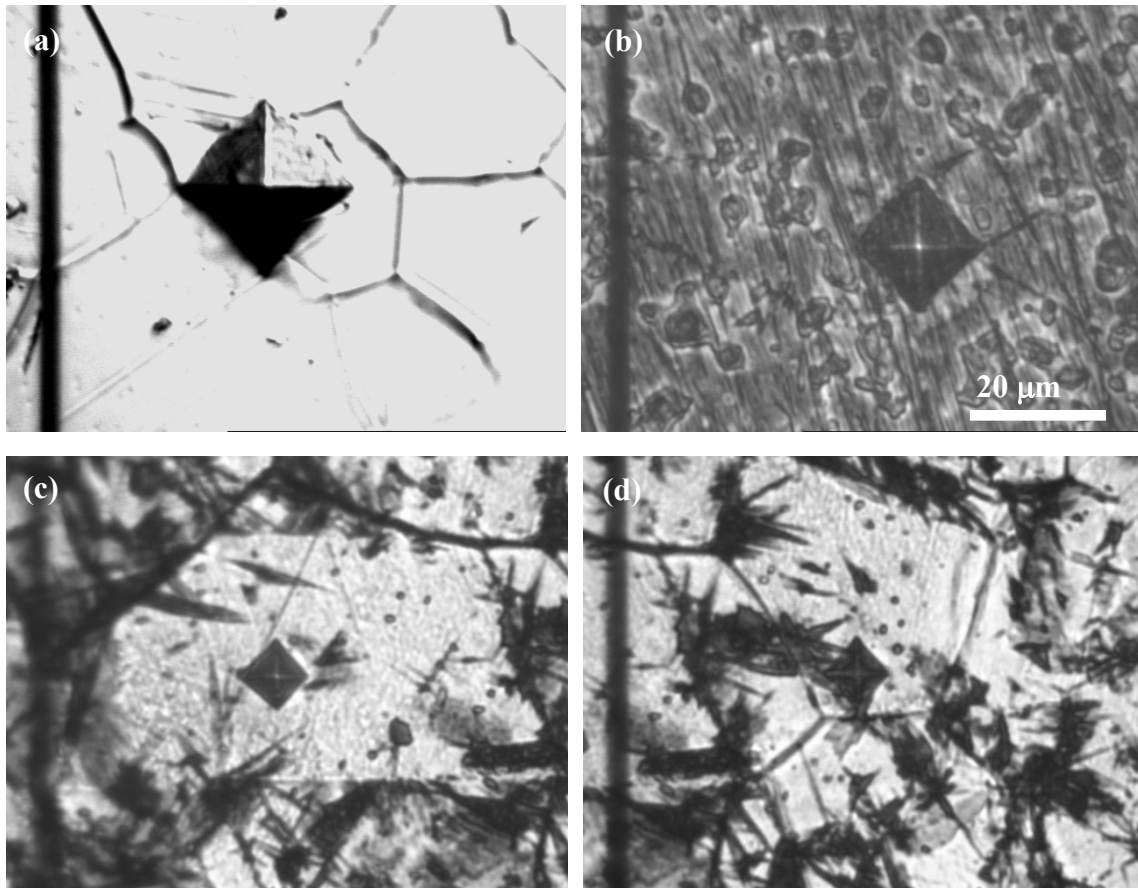


Fig. 2. Microstructures and indentation impressions in the annealed materials in (a) as-annealed, (b) annealed + irradiated, (c) annealed + Kolsterised, and (d) annealed + Kolsterised + irradiated conditions.

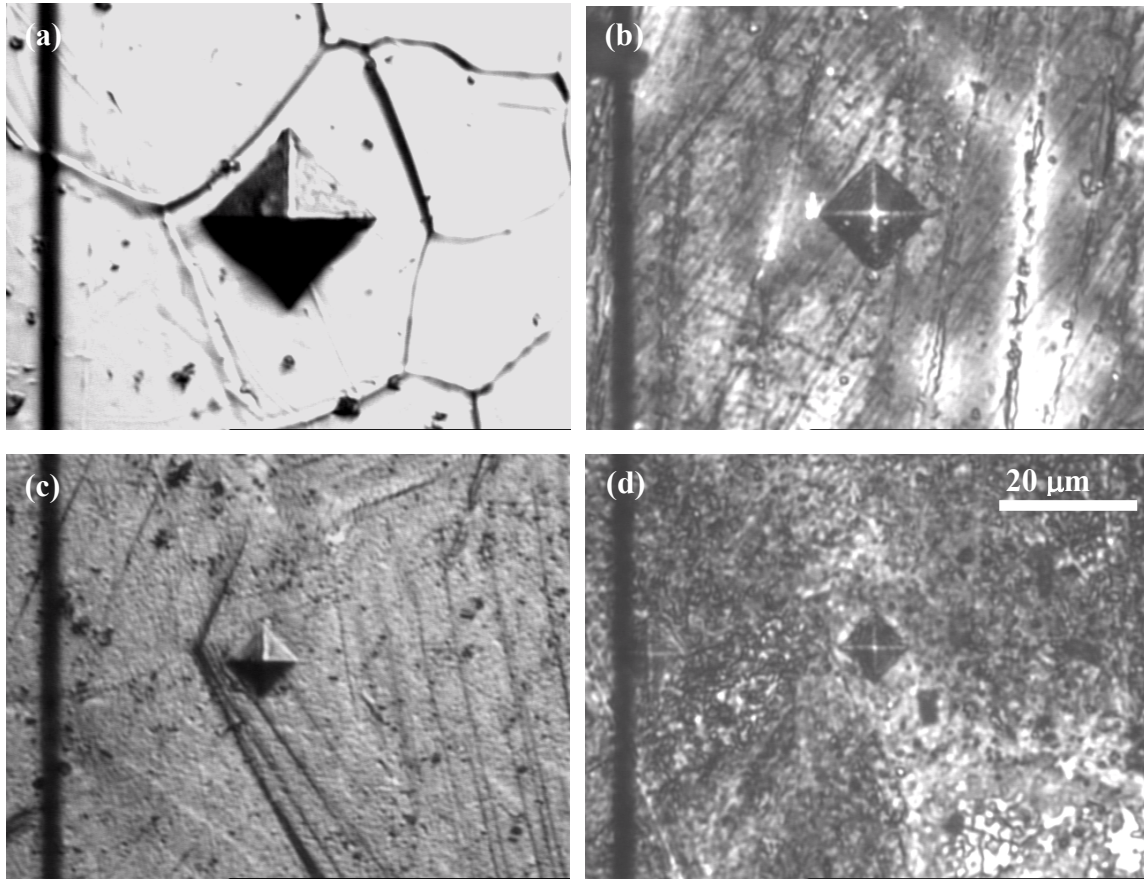


Fig. 3. Microstructures and indentation impressions in the 20% cold rolled materials in (a) as-rolled, (b) rolled and irradiated, (c) rolled and Kolsterised, and (d) rolled, Kolsterised, and irradiated conditions.

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