



The effect of applied compressive stress on the diffusion of carbon in carbon supersaturated S-phase layer



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ABSTRACT

In this study, hydrostatic compressive stresses are applied on the prior-carburized samples of 316 ASS to discover the relationship between interstitial diffusion process and the applied stresses. Carbon diffusion profiles are compared and concentration dependent diffusion coefficients are discussed to further advance scientific understanding of diffusion process in supersaturated S-phase. The experimental results demonstrate that the applied compressive stress can retard the fast diffusion process comparing to the unstressed one. Based on theoretical modelling, the effect of the applied hydrostatic compressive stresses on the diffusion of carbon in carbon supersaturated 316 austenitic stainless steel is discussed.

1. Introduction

S-phase, interstitial supersaturated expanded austenite, is produced on the surface of austenitic stainless steel by low temperature nitriding, carburizing or carbonitriding. It arouses great interests by performing extremely high hardness, excellent wear resistibility and brilliant fatigue property [1]. It is generally believed that S-phase is an interstitial supersaturated single phase without precipitates [2–4]. Hence, the most significant signature of S-phase, supersaturation, has been verified by many researchers [5–8] and the value of the interstitial supersaturation can reach about 12 at.% in carburized surface S-phase layer and about 25–30 at.% in nitrided ones [9–12].

The earlier prediction of nitrogen depth profiles in plasma nitrided ferrite steel containing nitride forming elements was developed by Sun and Bell et al. [13] and several models have been proposed to explain the tremendous uptake of nitrogen in S-phase. One is the trapping and detrapping model which was developed by Parascandola et al. [14] considering the assumption that diffusion of nitrogen is influenced by trap sites of local chromium. Another model by Mandl et al. [15] assumed that N diffusion in S-phase was concentration dependent, with high diffusion coefficients for high nitrogen contents and low value for low ones. Contrary to the trapping/detrapping model, Mandl et al. [16] studied different steel grades and found that there is no clear correlation between the critical point of nitrogen diffusion and the chromium content. Besides, Abrasonis et al. [17] explained the influence of flux on the ion-beam nitriding of austenitic stainless steel by considering free

diffusion-sputtering. Christiansen et al. [18,19] simulated the diffusion of N in austenitic stainless steel with different equilibrium constants. Recent work by Wu et al. [20] showed that the diffusivity difference between carbon and nitrogen increases with increasing Cr concentration but the carbonitriding results demonstrated that the diffusion depth of carbon or nitrogen is not affected by the presence of each other. Clearly, it is still a challenge to predict the depth distribution of interstitials in S-phase layers despite the great effort made by these researchers.

Meanwhile, an interesting issue kinked with the supersaturation of interstitials in S-phase is the large compressive residual stresses which was evaluated to be about -2.7 GPa for carburized S-phase and even larger for nitrided ones [21,22]. Although thermal stresses may be introduced upon cooling from the treatment temperature to room temperature, the residual stress is mainly induced by the supersaturated interstitials and vice versa the stress conditions can also influence the diffusion of interstitials [23]. Therefore, the diffusion of interstitial atoms in S-phase is complicated by the mutual effects of interstitial compositions and stresses. X-ray diffraction pattern in S-phase showed a shift of the (200) peak comparing with the untreated austenite probably because of stacking faults (SFs) created by cold working [24,25]. Both low temperature carburizing and nitriding show some interesting diffusion phenomenon. Although the effect of the in-situ applied tensile stresses on the formation of S-phase during low-temperature carburizing of 316 steel has been reported in our previous paper [26], to date, no work has been reported on the effect of external compressive stress

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Table 1
The chemical composition of 316 ASS (at.%).

C	Cr	Ni	Mo	Mn	Si	P	S	Cu	N	Fe
0.23	18.35	10.32	1.18	1.62	0.94	< 0.05	0.01	0.35	0.29	Balance

on carbon supersaturated expanded austenite i.e. S-phase.

In this study, in order to study the effect of external compressive stress on carbon diffusion in carbon supersaturated expanded austenite (i.e. S-phase), hydrostatic compressive stresses are applied through hot isostatic pressing (HIPping) to low-temperature carburised (i.e. carbon S-phase) 316 steel samples. Carbon depth-distribution profiles are carefully measured and compared; carbon diffusion process under the applied compressive stresses is analyzed; and concentration dependent diffusion coefficients are calculated. Finally, the effect of the applied compressive stresses on the diffusion of carbon in the carbon S-phase is discussed to further advance scientific understanding of interstitial diffusion in supersaturated S-phase under compressive stress.

2. Experimental

The material used in this study is 316 austenitic stainless steel with nominal composition shown in Table 1. The samples were cut from hot-rolled bars with diameter of 25 mm into 5.5 mm thick disks, using a high precision Struers Accutom-5 cutting machine, fitted with SiC abrasive cutting blades. Then the samples were wet ground into flat surfaces with SiC grinding paper of grit #120. The one face which was to be carburized was ground further up to grit #1200 (#240, #400, #800 in steps). Then samples were washed in soap water and acetone, both in an ultrasonic bath, for 10 min, then dried with blow-air.

Based on the optimal values identified by previous research [27], low temperature plasma carburizing treatment was carried out in a 60 kW Klöckner DC plasma unit at 500 °C for 10 h in 400 Pa gas mixtures with 1.5% CH₄ and 98.5% H₂. This will also facilitate the comparison with the earlier work on the effect of temperature on the stability of S-phase [23]. After plasma carburizing treatment (sample coded as 'PC'), samples were carefully taken out and swabbed with acetone to remove the superficial very thin deposition layer of amorphous carbon although Fe₃C and oxides were also identified by other researchers [28]. Then samples were separated into two groups and the group one samples were sealed individually in a vacuumed quartz tube with a few refilled argon gas. Then these samples were heated treated heated up to the conditions as listed in Table 1 in a Muffle furnace. For group two, samples were put into an EPSI Lab HIP (Hot Isostatic Pressing) facility and were heated up to designed temperatures with hydrostatic compressive stresses by argon gas for different time as detailed in Table 2. The group one samples were coded as 'HT', and the samples from group two were coded as 'HIP'. Limited by the capacity of the HIP equipment, short soaking time (1–10 h) and relatively high soaking temperature (525–600 °C) were chosen to promote diffusion process while suppress potential carbide precipitations.

Table 2
Sample codes and corresponding HIP and HT treatment conditions for plasma carburized (PC) samples.

Samples code	Temperature °C	Pressure MPa	Soaking time h
HIP/HT 1	525	180/0	10
HIP/HT 2	550	180/0	1
HIP/HT 3	550	180/0	5
HIP 4	575	90	1
HIP/HT 5	575	180/0	1
HIP/HT 6	575	180/0	2
HIP/HT 7	600	180/0	0.5
HIP/HT 8	600	180/0	1

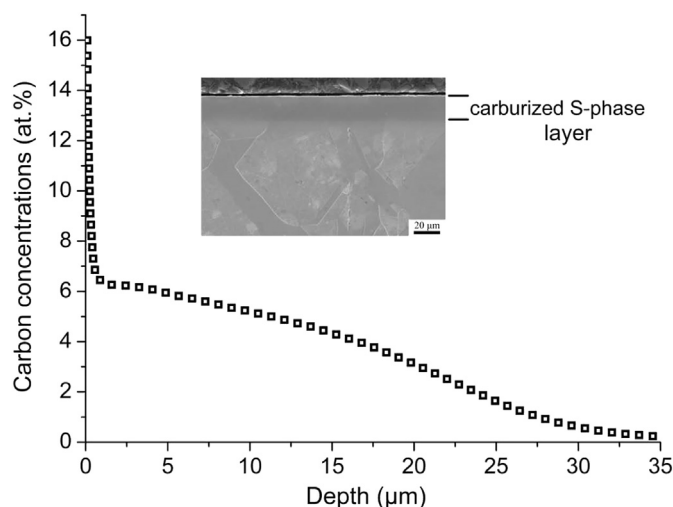


Fig. 1. Carbon depth profile of sample 'PC' prior to HIPping and annealing treatments. Cross sectional SEM image was inserted.

A Leco GDS-750 QDP unit was used to probe the composition-depth profiles of the carburized and tempered samples. In this technique surface atoms were sputtered from the sample and went into the argon plasma, where de-excitation process occurred and each species of particles would emit a characterized spectrum of light from which the quantity of these atoms could be evaluated from the intensity of their specified spectrum. The method and measurement program were calibrated using standard blocks of known composition, according to the instructions of the manufacturer. The measurement was repeated at least three times for each sample and the reproducible results were reported. Cross-sectional microstructures were observed by Jeol 7000 field emission gun (FEG) SEM after grinding and polishing, then etching with a 50% HCl + 25% HNO₃ + 25% H₂O etchant. A Philips X'Pert diffractometer was employed to obtain the x-ray patterns of samples using Cu radiation with K_{α1} 0.15406 nm.

3. Results

A typical carbon profile after carburizing treatment on 316 austenitic stainless steel is illustrated in Fig. 1. It is noteworthy that the highest amount at the top surface can reach above 12 at.%. Then there is a plateau at about 6.5 at.% before it gradually depleted into the substrate.

Fig. 2 shows the carbon depth profiles of samples 'HT 1' and 'HIP 1' comparing with 'PC'. The trend of carbon depth profiles revealed that the maximum carbon content near the surface was significantly reduced but the thickness of the surface case was increased after HIPping and HT treatments due to inward diffusion of carbon during these treatments. However, it can be deduced by comparing the carbon depth profiles in the 'HT 1' and 'HIP 1' samples that the compressive stress applied by HIPping treatment retarded the diffusion of carbon atoms from the carburized surface cases towards the central substrate. Consequently, the 'HIP 1' sample had a high carbon quantity in the top surface case of about 25 μm but a shallow carbon penetration into the substrate.

Similar phenomenon was observed for samples heated up to 550, 575 and 600 °C. This is further supported by the effect of compressive stress levels on the diffusion of carbon at 575 °C for 1 h. As shown in Fig. 3, the carbon depth profile of the 90 MPa HIPped sample ('HIP 4') was located in-between those of the 0 MPa ('HT 5') and 180 MPa ('HIP 5') treated samples. For the same diffusion time (1 h) but at different temperatures, the carbon diffusion profiles behave differently as shown in Fig. 4.

Fig. 5 show the XRD charts of HIP and HT samples treated at a

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