

# Effect of tensile stress on the formation of S-phase during low-temperature plasma carburizing of 316L foil

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## Abstract

Low-temperature plasma carburizing of austenitic stainless steel can produce a carbon-supersaturated austenite layer, the “S-phase”, on the surface, which has high hardness, excellent wear and fatigue properties, and good corrosion resistance. Although the S-phase was discovered some years ago, the basic understanding of S-phase formation remains incomplete. In this paper, the effect of tensile stresses (0–80 MPa) on the formation and stability of S-phase during carburizing of 316L stainless steel foils at 400, 425 and 450 °C for 10 h has been investigated for the first time. The microstructures were characterized by X-ray diffraction, scanning electron microscopy and transmission electron microscopy and the mechanical properties were evaluated by microhardness and tensile tests. The results showed that the in situ applied tensile stress effectively thickened S-phase layers. The calculated activation energy for carbon diffusion in 316L was reduced from 142.76 to 133.91 kJ mol<sup>−1</sup> when a tensile stress of 40 MPa was applied. However, chromium carbides were formed in the outmost surface when the tensile stress exceeded 40 MPa. The results are discussed and explained through appropriate thermodynamic calculations. © 2011 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

**Keywords:** Austenitic stainless steels; Plasma carburizing; S-phase; Stress-effect; Thermodynamics

## 1. Introduction

Due to its excellent corrosion resistance, adequate mechanical properties and good fabricability, AISI 316 austenitic stainless steel is used in a wide range of applications, from normal decorative material used in the kitchen and in furniture to working pieces in mechanical devices and machines, and critical components in the nuclear and space industries. However, it is well known that austenitic stainless steel is characterized by poor tribological properties, mainly because of its low hardness and strong adhesion tendency. Accordingly, austenitic stainless steel is normally limited to non-tribological applications [1].

Thermochemical surface engineering (such as carburizing and nitriding) has long been used to improve the hardness and tribological properties of many types of

metallic materials. However, conventional thermochemical treatments are not suitable for austenitic stainless steel because the increased hardness and wear resistance is achieved at the price of reduced corrosion resistance due to the precipitation of chromium carbides or nitrides and the depletion of chromium in solid solution. This technical challenge was not addressed until the mid-1980s, when interstitially supersaturated austenite, i.e. the so-called “S-phase”, was created in austenitic stainless steel by low-temperature thermochemical treatments [2].

It has been reported that the formation of S-phase on the outer surface can greatly enhance the mechanical properties of austenitic stainless steels, such as hardness, wear resistance and fatigue properties [3–6], because of the supersaturation by interstitials. Without the precipitation of carbides or nitrides, the good corrosion resistance of austenitic stainless steel can be maintained and in some cases even improved [7].

One of the technological challenges associated with S-phase surface engineering is that the maximum layer

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Table 1  
Chemical composition of AISI 316L stainless steel (at.%).

| C    | Cr    | Ni    | Mo   | Mn   | Si   | P     | S    | Cu   | N    | Co   | Al   | Fe      |
|------|-------|-------|------|------|------|-------|------|------|------|------|------|---------|
| 0.15 | 18.76 | 10.65 | 1.10 | 1.61 | 0.98 | <0.05 | 0.01 | 0.35 | 0.24 | 0.19 | 0.21 | Balance |

thickness of the S-phase layers is much thinner than that formed during the nitriding and carburizing of other steels [2]. This is because S-phase is thermodynamically metastable and the precipitation of carbides or nitrides will occur as a result of prolonged treatment. For example,  $\chi$ -carbide ( $M_5C_2$ ) was found after prolonged gas carburizing treatment at 748 K for 44 h on 316L stainless steel [8]. Clearly, how to increase the maximum layer thickness of S-phase without precipitation is an technologically important research topic in S-phase surface engineering. Extremely high residual stresses for carburizing and nitriding were found to exist in the S-phase [9] and it is envisaged that applying a tensile stress during S-phase formation could increase the thickness of S-phase layers.

The study of stress on the formation of S-phase also has real value in some engineering applications, such as the formation of S-phase on vertically suspended long tubes for nuclear reactor components [10], in which tensile stress is inevitable because of its own weight. However, the effect of tensile stress on the formation of S-phase and potential precipitation of chromium carbides or nitrides is almost absent from the literature. This may be crucial for critical nuclear reactor components since the formation of chromium carbides or nitrides will dramatically reduce their corrosion resistance, which could lead to serious safety problems.

Finally, investigation into the effect of in situ applied stress on the formation of S-phase, the decomposition of S-phase and the precipitation of carbides or nitrides could advance scientific understanding of the diffusion of interstitials, the metastability of S-phase and the mechanism of S-phase formation.

Accordingly, the present investigation was directed at identifying and understanding the effect of applied tensile stress on the formation of S-phase on AISI 316L stainless steel during low-temperature plasma carburizing with the aims of: (i) exploring the possibility of increasing the S-phase layer thickness by applying tensile stress during treatment; (ii) studying the decomposition of S-phase under tensile stress; and (iii) advancing scientific understanding of the mechanism of S-phase formation and diffusion of interstitials under tensile stress. To this end, the effect of stresses on the formation of S-phase during carburizing of AISI 316L stainless steel foils was investigated for the first time by applying a constant stress. The microstructures were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM), and the mechanical properties were evaluated by microhardness and tensile tests. The results are discussed and explained through appropriate thermodynamic calculations.

## 2. Experimental

### 2.1. Material

The material used for study is AISI 316L stainless steel in the form of thin foil, 50  $\mu\text{m}$  thick and 1 cm wide. All samples were washed in soapy water and then acetone in an ultrasonic tank before treatment. The chemical composition of 316L stainless steel is listed in Table 1.

### 2.2. Plasma carburizing treatments

A series of experiments were designed to study the effect of stress on the formation of S-phase in terms of fixed plasma carburizing conditions under different in situ tensile stresses and, conversely, under a constant in situ stress with plasma carburizing at different temperatures and times in a fixed atmosphere. The experimental equipment for plasma carburizing under tensile stress is shown schematically in Fig. 1. 316L stainless steel foils were suspended from a supporting frame and different loads were added to the bottom side prior to the plasma carburizing treatment to produce a constant tensile stress. The carburizing treatments were carried out in a 60 kW Klöckner DC plasma vacuum unit in a 400 Pa gas mixture of 1.5%  $\text{CH}_4$  and 98.5%  $\text{H}_2$ .

As summarized in Table 2, the first batch of treatments at 450 °C for 10 h used tensile stresses from 0 to 80 MPa

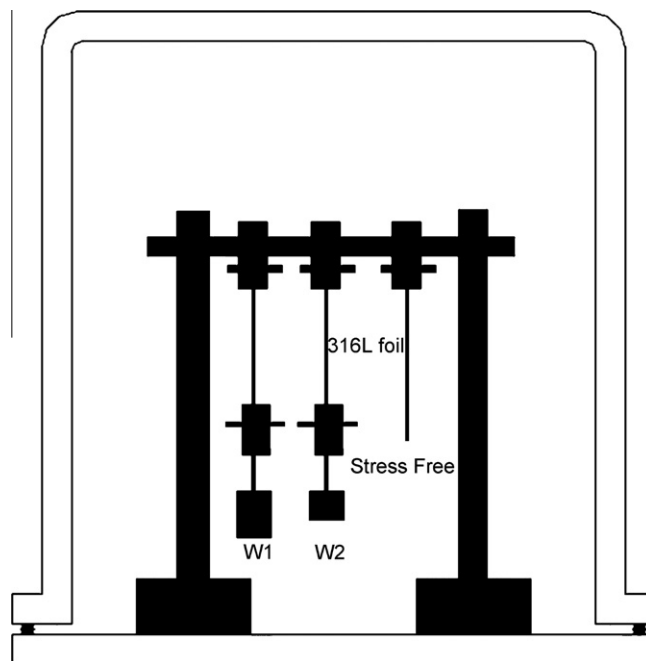


Fig. 1. Schematic drawing of the in situ stressing installation in a plasma carburizing furnace.

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