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Diffusion behavior of carbon and its hardening effect on plasma carburized M50NiL steel: Influences of treatment temperature and duration

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ABSTRACT

Diffusion behavior of carbon and its hardening effect on plasma carburized M50NiL steel were investigated systematically. The plasma carburizing was carried out in a mixture of gaseous acetone (30%) and hydrogen (70%) at temperatures of 400, 450, 500 and 550 °C for durations of 4, 8 and 12 h, respectively. Influences of treatment temperature and duration on properties of the carburized layer were investigated with optical microscope, atomic force microscope, X-ray diffractometer, micro- and nano-indenters, and pin-on-disc tribometer. Results of the tests indicate that cementite is the main phase in the surface layer of specimens carburized at 400 and 450 °C, while that in those carburized at 500 and 550 °C is mainly the carbon expanded martensite. The specimens with cementite in the surface layer show lower surface roughness, higher surface hardness and thus lower wear rate. The diffusion behavior of carbon and resultant microstructure evolution mechanism were studied, indicating that plasma carburizing is a diffusion-controlled process. Activation energies of carbon diffusion into martensite and cementite were calculated, which are 64.5 kJ·mol^{-1} and 120.7 kJ·mol^{-1}, respectively. The information on the activation energies provides basic data for tuning and predicting the surface properties of plasma carburized M50NiL steel.

1. Introduction

As an efficient and environmental friendly treatment, plasma carburizing provides an effective process to improve surface properties of steels. This process is particular attractive for stainless steel [1–5], since the involved sputtering helps to remove the native oxide film (Cr_2O_3) on stainless steel, which generates a diffusion barrier and reduces the adhesion between a protective coating and the steel surface [5]. Furthermore, as reported in the literature [1–5], low-temperature plasma carburizing is very promising to produce high-quality modified layer with high resistance to wear and corrosion simultaneously.

M50NiL steel has a composite microstructure with case-hardened surface and a relatively tough core after surface treatment, which meets the requirements for aircraft engine components that work under arduous aircraft engine conditions [6–8]. Traditional high-temperature gas carburizing gives rise to favorable compressive stress in the surface layer, which endows M50NiL bearings a higher resistance to rolling contact fatigue. However, it is giving way to low-temperature plasma carburizing due to its limitations related to performance, reliability and economy for future aircraft engines [9]. For instance, the high temperature process leads to coarse microstructure, severe deformation and high energy consumption. In our previous work [10], low-temperature plasma carburizing for M50NiL steel in different gas mixtures was investigated, showing its great promise as a surface treatment method for M50NiL steel. However, the information on influences of treatment temperature and duration on properties of carburized layer of M50NiL steel is unavailable. It is thus of importance to investigate how these two process parameters affect the microstructure and properties of plasma carburized M50NiL steel.

The dynamics of carbon diffusion in steel affects the microstructure evolution during the carburizing treatment. The activation energy, a crucial thermodynamic parameter, is essential to process design and control, since it (a) influences the layer thickness with respect to temperature and time, and (b) provides clues for determining an adequate process condition for effective carburizing [11]. Scheuer et al. [2] systematically investigated the low-temperature plasma carburizing kinetics for AISI 420 stainless steel and calculated the activation energies for compound and diffusion layer growth. Their results indicate that low-temperature plasma carburizing is a diffusion controlled process. However, the calculated activation energy (29 kJ·mol^{-1}) for the compound layer is too low, compared to the theoretical value (109 kJ·mol^{-1}) for carbon diffusion in cementite. There are

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discrepancies between experimental observations and theoretical predictions. Thus, it has become an urgent and significant task to systematically investigate the diffusion behavior of carbon during the plasma carburizing based on experimental results for effective process control and design.

In the present work, the influences of treatment temperature and duration on microstructure, phase composition and mechanical properties of M50NiL steel were investigated. The diffusion behavior was analyzed based on microstructure evolution, and the activation energies of carbon diffusion into martensite and cementite were calculated. Results of the study would provide helpful information for predicting and controlling surface properties and layer growth in plasma carburized steels.

2. Experimental procedure

2.1. Materials and experimental methods

The as-received M50NiL steel under study contains the following elements, 0.13C, 4.1Cr, 3.4Ni, 4.2Mo, 1.2V, 0.13Mn, 0.18Si, 0.012P, 0.002S and balanced Fe (wt%). The steel was solution treated at 1050 °C for 1 h followed by gas quenching. Prior to the plasma treatment, steel specimens with 12 mm × 12 mm × 5 mm dimensions were polished using SiC papers (from 240 to 800 grade).

The specimens were carburized in a pulsed glow discharge plasma unit (LDMC-30, 30 kW) [12]. Sputtering pre-treatment using hydrogen under a pulsed dc voltage (U = 660 V) was applied to clean and activate the specimen surface during the heating-up stage. Plasma carburizing was carried out with a gas mixture containing gaseous acetone (0.15 L/min), hydrogen (0.35 L/min). Gaseous acetone, which acted as the source of active carbon atoms, was obtained by heating the pure acetone at 40 °C. According to previous studies [2,13], the critical temperature for different diffusion kinetics in plasma carburizing is in the range of 450–500 °C. Thus, for the present work, the specimens under study were treated at 400, 450, 500 and 550 °C for 4, 8 and 12 h, respectively. After the treatment, the specimens were directly cooled to room temperature in the furnace.

2.2. Characterization

Cross-sections of the modified layer were polished and etched in a 4% Nital solution for optical examination with an OLYPUS PME3 optical microscope (OM). The surface morphology and roughness were analyzed under an atomic force microscope with a nano-Kelvin Probe (MAFM, Brucker Multimode 8). The phase structures in the modified surface layer and subsurface layer (at 10 μm distance from surface) were analyzed using X-ray diffractometer (XRD, type D/max-rB) with Cu-Kα radiation (λ = 0.15406 nm) in the range of angles 20–100°. Nano-hardness and Young's modulus of the surface were measured using a nano Indenter (XP system, MTS) in the continuous stiffness mode (CSM), and the indentation depth was 1 μm. Five indentation tests were performed, from which the mean value was calculated. Micro-hardness of the modified layer was measured at locations from 2 μm underneath surface to the core using a Vickers hardness tester (type HV-1000) under a load of 100 g with a dwelling time of 15 s. Three indentations for each indentation depth were made and the average value was calculated. The layer depth was determined based on microhardness profile, and the value equals to the depth for which the hardness is 50 HV more than the bulk material. Dry wear properties of the specimens were evaluated using a pin-on-disc tribometer (type POD-1). During the wear test, the specimen was rotated against a stationary WC ball of 5 mm diameter at speed of 200 r·min^{-1} (0.1 m·s) for 5000 s under a load of 20 N. The wear rate η (mm^3·N^{-1}·m^{-1}) was calculated using the following equation,

$$\eta = \frac{V}{FL} \tag{1}$$

where F (N) is the normal contact load and L (m) is the total sliding distance. V (mm^3) is the volume loss expressed as

$$V = \pi DS \tag{2}$$

where D (mm) is the diameter of the circular wear track. S (mm^2) is the cross-sectional area of the wear track measured using a stylus profilometer (CCI MP, Taylor Hobson).

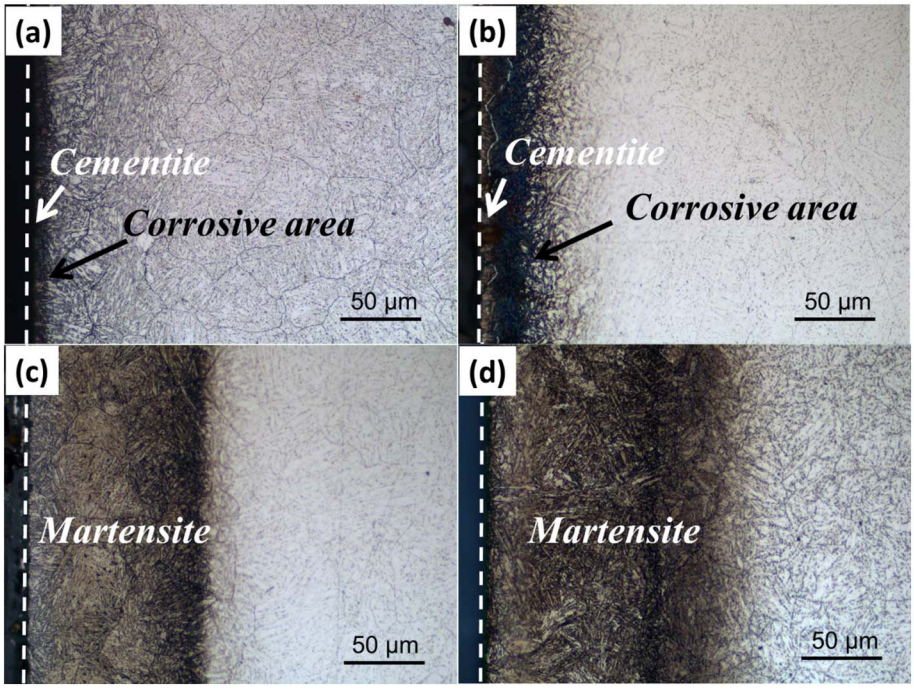

Fig. 1. Optical micrographs of specimens treated at different temperatures for 8 h: (a) 400 °C, (b) 450 °C, (c) 500 °C, (d) 550 °C.