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Surface nanostructure and improved microhardness of 40CrNiMo7 steel induced by high current pulsed electron beam treatment

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ABSTRACT

In this paper, surface modification of 40CrNiMo7 steel was investigated with high current pulsed electron beam (HCPEB) treatment. The scanning electron microscope (SEM), electron back-scattered diffraction (EBSD), electron probe micro analysis (EPMA), transmission electron microscopy (TEM) and X-ray diffraction (XRD) results show that a composite microstructure of mainly refined austenite and a little martensite was produced in the surface modified layer of depth ~7 μm. The average size of small cells on modified surface was decreased to ~120 nm after 25 HCPEB pulses. XRD analysis indicates a preferred orientation of austenite (220) crystal plane, and TEM results show the broken and dissolved cementite in the surface modified layer. After HCPEB treatment, all the samples exhibited a remarkable improvement in surface microhardness measurement, up to ~1000 HK for 15 HCPEB pulses, as tripled of the initial 40CrNiMo7 steel.

1. Introduction

Surface nanocrystallization has been proven to be an effective approach to improve surface engineering properties of industrial components. To date, various kinds of methods have been proposed based on surface deformation processes including surface mechanical attrition treatment (SMAT) [1], high-energy shot peening (HESP) and ultrasonic surface rolling processing (USRP) [2], and energetic beam irradiation techniques, such as laser, ion, electron beam and plasma flow treatment [3–5]. Compared with other competing techniques, HCPEB treatment has achieved a rapid progress as an effective method for surface modification of materials. During the HCPEB treatment, incident electron flux of extremely high energy (~30 kV) transfers into material surface (several micrometers) within a few microseconds. Consequently, different physical processes such as melt or evaporation are caused by the rapid heating (10^{9-10} Ks^{-1}), meanwhile, self-quenching (10^{8-9} Ks^{-1}) and thermal stress impact processes occur quite intensively in the near surface layers. Thus, the non-equilibrium surface microstructure with modified surface properties would be produced. Besides, the HCPEB treatment has significant advantages in reliability, efficiency and low cost [6–14].

As a typical high strength structure steel, 40CrNiMo7 steel has good balance of strength, toughness and fatigue property, and it has been widely adopted in critical structural applications, such as spindles, turbine rotor, automotive crankshaft, aircraft landing gear, etc. This paper aims to investigate the surface microstructure evolution of 40CrNiMo7 steel under HCPEB treatment, thereby exploring an efficient approach to improve the surface mechanical property of high strength structure steels.

2. Experimentation process

Shenyang Blower Group Co., LTD provided the 40CrNiMo7 steel ingot with the chemical components (wt%) displayed in Table 1. The cubic samples were obtained from the ingot with the size of 10 mm × 8 mm × 10 mm. Prior to HCPEB treatment, all the surface of samples was properly grounded, polished and cleaned ultrasonically in acetone. The surface treatment experiment was conducted using type HOPE-I HCPEB apparatus [15,17]. The treating parameters were as follows: accelerating voltage 27 kV, vacuum pressure 6×10^{-3} Pa, energy density 3 J/cm^2, pulse duration 2.5 μs and treating pulse 1, 6, 15 and 25.

The initial surface microstructure (etched by aquaregia mixed of HCL and HNO_3 acids with volume ratio 3:1) was observed by optical microscopes (OM) of type Olympus BX51. After HCPEB treatment, the microstructure characteristics of 40CrNiMo7 steel was observed using OM of type Olympus GX51, scanning electron microscope (SEM) of type Zeiss super 55 with an electron back-scattered diffraction (EBSD) attachment, and transmission electron microscope (TEM) of type JEM-2100F (HR). One-side milling and

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Table 1
Chemical composition of 40CrNiMo7 steel (wt%).

C	Cr	Ni	Mo	Mn	Si
0.37–0.43	0.60–0.90	1.60–1.90	0.20–0.35	0.50–0.80	0.15–0.40

single-jet thinning techniques were applied to prepare the thin foils for TEM observation. For the EBSD analysis, the recording and indexing of the pseudo-Kikuchi lines were made with HKL Channel 5 software. Electron probe micro analysis (EPMA)-1600 was used to applied the component distribution of cross-sectional morphology. Bruck D8 X-ray diffraction (XRD) with CuKα radiation was adopted to examine the change of phase structure on the modified surface, with scanning range (2θ) 20°–100° and step size of 0.04°. The DMH-2LS Knoop tester was used to measure the surface microhardness (HK), and the applied load was 10 g.

3. Results and discussion

3.1. Surface and cross-sectional morphology

Typical surface observation results are indicated in Fig. 1. The initial 40CrNiMo7 steel after normalizing process was composed of pearlite (dark contrast) and ferrite phases with average grain sizes of 40 and 60 μm, respectively (Fig. 1a). In the very first HCPEB treatment, as shown in Fig. 1b, the craters formed preferentially in the pearlite zone due to the relative lower melting point (1227 °C) and the large amount of interlayer structures between ferrite and cementite phases. It has been commonly believed that the eruption of crater is aroused by non-equilibrium thermal stress reinforcement around the structural defects in surface layer being treated by HCPEB. Fortunately, the craters could be healed by more successive pulses treatment [7–13]. Besides, as shown in Fig. 1c, many embossed lamellar structures appeared in the modified surface, which should be associated with the martensite phase transformation processes due to the super-fast cooling speed under the HCPEB treatment. Fig. 1d gives high magnification SEM morphology. All the lamellar structures were covered with a layer composed of small cells of average size ~100 nm. This kind of nanostructure obtained through high-speed crystallization of melt and with very little disorientation was named nanocrystalline cells in the following discussions. Similarly, after 6, 15 and 25 pulses HCPEB treatment, the nanocrystalline cells were observed on the treated surface, as shown in Fig. 1e to g. With the increasing pulse number of HCPEB treatment, the average cell size in the covering layer firstly increased to ~200 nm for 6 pulses, and decreased to ~170 nm for 15 pulses and then ~120 nm for 25 pulses. For the beginning pulses, the superfast cooling rate leads to the formation of nanocrystalline cells microstructure. With the increasing pulse number of HCPEB treatment, the cooling rate in surface layer would decrease gradually, in addition, the dissolution of C element was reinforced and the composition homogenization could be realized. These two competitive factors act simultaneously and give rise to the variation of crystallization size on HCPEB modified surface. Fig. 1h is the EBSD phase map image measured on the top surface of sample after 25 HCPEB pulses treatment. The surface compositions detected by Kikuchi patterns were indexed by Fe-BCC (martensite), Fe_3C and Fe-FCC (austenite) phases denoted by red, blue and yellow colors respectively. According to the phase percentage calculations, the austenite phase became the main part on the modified surface after 25 HCPEB pulses treatment. Referring to the SEI image, the composite nanostructure was produced on the HCPEB modified surface.

Fig. 2a shows the typical cross-sectional OM morphology of 40CrNiMo7 steel after HCPEB treatment. The graded modified microstructure was formed clearly along the HCPEB incident direction. The outermost layer was the remelted layer with a fairly homogeneous composition. Besides, the heat-affected zone (HAZ) and the matrix located under the remelted layer. The thickness of modified layer including remelted layer and heat-affected zone reached ~7 μm.

The EPMA was conducted on the cross-section of 40CrNiMo7 sample as shown in Fig. 2b. It is observed that in the substrate matrix, the average carbon content of ferrite is 60 (counts) and that of perlite is 180 (counts), while, the average carbon content in the surface modified layer is 90 (counts), which is 67% less than that of perlite and 50% more than that of ferrite. It could be concluded that the surface repetitive remelting produced the diffusion of carbon element and its homogenization in the surface modified layer.

3.2. XRD analysis and TEM observation

Fig. 3 gives the phase composition change in the surface layer of 40CrNiMo7 steel after HCPEB treatment by XRD, where the normalization procedure was applied to illustrate the peak intensity evolutions. For the initial sample, the sharp diffraction peaks were coincided well with the single ferrite (α-Fe) phase composition, while the diffraction peaks of cementite (F_3C) can be hardly identified by XRD method due to its relative low volume fraction. After 1 pulse of HCPEB treatment, all the diffraction peaks became blunt and broaden, furthermore, the peaks of α-Fe (200) and (211) were splitted. Based on the microstructure observation results above and PDF data of martensite (α′-Fe) as denoted, it is clear that the rapid melting and cooling processes associated with HCPEB treatment will induce the formation of greatly refined grains and the martensite phase transformation in the modified surface of 40CrNiMo7 steel. And a new diffraction peaks were detected in the XRD traces corresponding to austenite (γ-Fe). Subsequently, the intensity of γ-Fe peaks kept increasing with the increased HCPEB pulses number. To illustrate this tendency more clearly, relative intensity of I_γ (200) peak in XRD patterns of 40CrNiMo7 steel after HCPEB treatment with different pulses was given in Table 2. Obviously, the relative content of γ-Fe increased gradually with the more HCPEB pulses. At last, the modified surface was mainly covered with γ-Fe phase. In addition, as shown in Table 3, peak intensity ratio of I_γ (200) and I_γ (220) in XRD pattern for the sample treated by HCPEB 25 pulses deviated significantly from PDF data. The preferred orientation of γ-Fe (220) plane was also observed.

The further microstructure changes in surface modified layer was analyzed by TEM as shown in Fig. 4, which is consistent with XRD and EBSD results. After 1 HCPEB pulse (Fig. 4b), the cementite (F_3C) of black contrast in pearlite phase became thinner as compared with the initial sample (Fig. 4a), and some parts were bent and broken. The typical lamellar martensite microstructure is shown in Fig. 4c, and it indicated that the rapid cooling process