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Plasma boriding of high strength alloy steel with nanostructured surface layer at low temperature assisted by air blast shot peening

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ABSTRACT

Plasma boriding of high strength alloy steel (AISI H13) with nanostructured surface layer fabricated by air blast shot peening(ABSP) was performed at $580\,^{\circ}$ C for 4 h. A continuous coating layer with thickness of about 4 μ m was composed with two phases of Fe₂B and FeB, and the nanohardness of borided layer was as high as 20 GPa. These results indicate that cyclic deformation and the angle between shot jet and sample surface play a critical role in the process to produce nanocrystalline in the surface layer. By using ABSP as pre-treatment, boron diffusion and the nucleation rate of boride can be remarkably enhanced in nanostructured surface layer. In addition, the weight loss rates of borided samples in molten aluminum alloy were only as much as ~40% of that of untreated ones, and the main reason was that the coating layer can effectively prevent the substrate directly contacting with molten aluminum alloy.

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1. Introduction

The AISI H13 steels are used extensively for extrusion dies as well as die casting tool steels for aluminum alloy and many other metal workpieces. They are alloy steels with high strength and toughness. However, this kinds of die steels is commonly used in aggressive environments, and it is necessary to adopt surface treatment technique to improve their surface properties, such as wear resistance, hardness and corrosion resistance. Boriding technique has attracted extensive attention in thermochemical surface treatment, owning to the borided layers possess excellent adhesion to the substrate when compared to prevalent physical coating process. The boriding process can be carried out in solid, liquid or gaseous medium [1]. Many previous literatures have reported the boriding techniques which are used to prepare borided layers on the surface of steels [2–5]. Yet, most of them have the disadvantages of requiring relative high processing temperature (750–950 °C) or time consuming, which are harmful to the mechanical properties of the matrix and create the deformation of workpiece. For H13 steels, a critical requirement for the boriding process is that it must be conducted at temperatures below 650 °C in order to prevent grain growth and carbide precipitation and hence to preserve their high strength and toughness. Therefore, boriding at lower temperatures and in shorter time have attracted extensive significant interest over the past decades. Plasma boriding was expected to be able to accelerate boron atoms diffusion into the steels, due to the nature of this method. It was regarded to be carried out at lower temperatures [6,7]. However, to date, reports on the plasma boriding that is used to synthesize borided layers on the surface of steels below 650 °C are scarce. In addition, little work has been focused on the study of dynamic erosion resistance properties of H13 die steels with plasma boriding treatment.

It is well known that borided layers generated in thermochemical treatment depends on boriding condition and on the properties of the materials itself. Both factors are strongly affected by grain boundaries and defect densities. The diffusion dynamics of boron atoms can be sufficiently enhanced in materials with nanostructured surface layer, due to the large grain boundaries and defect densities. Various severe plastic deformation methods have been proposed to produce nanocrystalline materials. Among these techniques, shot peening treatment can not only lead to residual compressive stress, but also fabricate ultrafine grain on the surface layer. Nanostructured surface layer can be obtained on stainless steel [8], low alloy steel [9], and silicon steel [10] by using air blast shot peening(ABSP), which has been applied extensively in material processing. But until now, high strength alloy steels with nanostructured surface layer through this method has not been reported. In this study, plasma boriding was carried out at low temperature for AISI H13 steels assisted by ABSP. Moreover, dynamic erosion resistance properties of them in molten aluminum alloy were investigated.

2. Experimental

2.1. Materials and shot peening treatment

The chemical compositions of AISI H13 steel used in the experiments contain (wt.%) 0.42C, 4.93Cr, 1.40Mo, 0.98Si, 0.87 V, 0.30Mn, 0.018P, 0.005S and balance Fe. The ferritic steel was pretreated by quenching

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at 1030 °C and tempering twice at 610 °C to obtain a martensitic microstructure. The hardness of sample was 46–47HRC. Before ABSP, the sample with the shape (60 mm \times 60 mm \times 4 mm) was mirror polished. Then, the sample was processed by a flow of cast steel balls with diameter of 0.8 mm at 0.5 MPa for six cyclic deformation, and the time of each cycle was 5 min.The angle between the shot jet and the sample surface is in the range of 70–90°. The small samples with dimensions of 15 mm \times 15 mm \times 4 mm for plasma boriding treatments were machined from the bulk sample as mentioned above. The specimens were ultrasonically cleaned with alcohol and acetone before plasma treatment.

2.2. Plasma treatment

The plasma boriding system mainly consisted of vacuum chamber equipped with electrical power and gas flow control units. After loading the small sample, it was cleaned by sputtering with a direct electrical power of 520 V in a gas mixture of Ar and H_2 . At the same time, the temperature of vacuum chamber increased to 580 °C with a heating rate of 2 °C/min. BF_3 was fed into the vacuum chamber with a certain flow rate. The gas consisted of BF_3 (3% vol.%), Ar (37% vol.%) and H_2 (60% vol.%). During our experiments, the total pressure had been maintained between 650 and 700 Pa for 4 h. The voltage applied on the samples was about 550 V.

2.3. Dynamic erosion measurements

Plasma boriding treatments were carried out for cylindrical samples with dimensions of 10 mm in diameter and 60 mm in length, which were used in the dynamic erosion tests. These samples were compared with the untreated ones. As the H13 steels are used extensively in die-cast formation for aluminum alloy, their dynamic erosion resistance to a molten aluminum alloy is very important. ADC12 aluminum alloy is a kind of die-casting aluminum which is widely used in industrial production. In order to study the performance of dynamic erosion resistance of borided samples and untreated ones to a molten aluminum alloy, ADC12 aluminum alloy was used in the dynamic erosion measurements. The chemical compositions of ADC12 aluminum alloy contain (wt.%): 2.13Cu, 0.87Fe, 10.72Si, 0.31Mg, 0.36Mn, 0.85Zn, 0.054Ti, 0.054Ni, 0.056P, 0.037Sn and balance Al. The proper quantities of ADC12 aluminum alloy was put into an alumina crucible and heated. When the melt temperature was stabilized at around 700 °C, borided sample and untreated one were held symmetrically on a rotating shaft driven by a DC electric motor and dipped into the molten aluminum alloy. The rotating shaft rotated at a speed of 120RPM. The schematic diagram of experimental set up used for dynamic erosion test system is illustrated in Fig. 1. The erosion studies were carried out for 5, 10, 15 and 30 min, respectively. Before measuring the weight loss rate, the aluminum which existed on the surface of specimens was removed with a NaOH solution and thoroughly rinsed with water, and the specimens were dried in an oven at 60 °C for 4 h.

2.4. Characterization

The deformation layer and nanostructured features obtained by ABSP were observed using a JSM-6301F scanning electron microscope (SEM)and a field emission transmission electron microscopy(TEM, JEM-2010F), respectively. The crystalline structure of samples was characterized with a RigakuD/Max-RBX-ray diffractometer by using CuKa radiation and a secondary beam graphite monochromator. The hardness gradient of nanoindentation test by using a Berkovich diamond indenter with a tip radius of about 200 nm was performed on Triboindenter In-Situ Nanomechanical Test System.

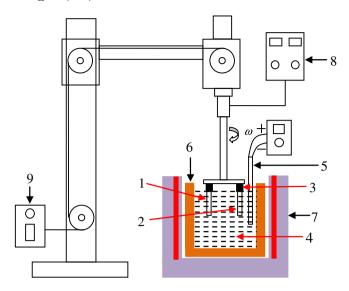


Fig. 1. Schematic diagram of experimental set up used for dynamic erosion test system: 1 borided sample, 2 untreated sample, 3 graphite sleeve, 4 ADC12 aluminum melt, 5 thermocouple, 6 alumina crucible, 7 salt bath furnace, 8 rotational speed control system. 9 electromotor.

3. Results and discussion

3.1. Characterization of the nanostructured surface layer

The SEM image of the cross section of the sample treated by ABSP is shown in Fig. 2(a). It can be seen that a plastic deformation layer $(\sim\!10\;\mu m)$ is present in the treated surface layer, which is indicated by a red-red line, where the microstructural morphology differs from that in the matrix. The boundary between deformation layer with dark contrast region and substrate is not distinctive. In subsurface zone, it is work-hardened region, which is characterized with the elongated grain boundaries. Fig. 2(b) displays a typical bright-field TEM image of the top layer and the statistical distribution of grain size(inset). The size of vast majority of grains is at 5–15 nm. The selected-area electron diffraction(SAED) pattern is shown in Fig. 2(c). It reveals that the microstructure is characterized by ultrafine equiaxed grains with random crystallographic orientation. The strong diffraction rings are assigned to polycrystalline α -Fe. There is also one reflection that could be interpreted as austenite reflection(311). It may reveal that α to γ transformation in the nanostructured surface layer at about room temperature during severe plastic deformation, due to the formation of supersaturated nanocrystalline ferrite and the compression stress at the boundaries of nanosize grains [11]. There is no carbides diffraction ring that can be detected, which may be attributed that the carbides might be broken into ultrafine particle and they are too small to be detected. Alternatively, they dissolved into the ultrafine ferrite matrix on the top surface layer where very large strain and strain rate were applied during the repetitive impacts of a lot of balls [12]. Fig. 2(d) shows the HRTEM image of the top surface layer. There is one elongated grain (indicated by dashed line) with the short axis of ~20 nm, which contains a subgrain boundary. It is composed of several edge dislocations. The elongated grain could be further refined with deformation increment.

The production of nanocrystalline surface layer by ABSP has considerable importance since ABSP is a popular process in industries. According to the previous report [13], there are some necessary and favorable deformation conditions to produce nanostructured surface layer on metal materials. Firstly, the minimum amount of strain necessary to produce nanocrystalline structure is considered to be around 7–8, depending on the deformation techniques and materials employed. Secondly, the nanostructured surface layer could be produced in the range of strain

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