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ВЛИЯНИЕ РЕДКОЗЕМЕЛЬНОГО $CeCl_3$ НА МИКРОСТРУКТУРУ И АДГЕЗИЮ БОРИДНОГО СЛОЯ ДЛЯ СТАЛИ AISI H13

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Аннотация. В данном исследовании сталь AISI H13 была подвергнута борированию редкоземельным композитом с использованием процесса пастообразного борирования. Были проанализированы структура и морфология боридного слоя, а также протестированы его твердость и адгезия к поверхности раздела. Результаты показывают, что слой борида имеет плотную пилообразную морфологию с минимальным количеством пор. По сравнению с боридным слоем без добавления $CeCl_3$ толщина восстановленного боридного слоя увеличилась с 53,7 мкм до 66,3 мкм. Кроме того, боридный слой из редкоземельного композита продемонстрировал твердость в диапазоне 1200–1700 HV с небольшим градиентом твердости. При испытании на адгезию наблюдалось лишь незначительное отслоение слоя борида редкоземельных элементов, что позволяет предположить, что редкоземельные элементы эффективно снижают хрупкость боридного слоя и улучшают адгезию к поверхности раздела.

Ключевые слова: сталь AISI H13, борирование композита, микротвердость, адгезия.

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Original article

EFFECT OF RARE EARTH $CeCl_3$ ON THE MICROSTRUCTURE AND ADHESION OF BORIDE LAYER FOR AISI H13 STEEL

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Abstract. In this study, AISI H13 steel was subjected to rare earth composite boronizing using the paste boronizing process. The structure and morphology of the boride layer were analyzed, and its hardness and interface adhesion were tested. The results indicate that the RE-boride layer exhibits a dense sawtooth morphology with minimal pores. Compared to the boride layer without the addition of CeCl_3 , the thickness of the RE-boride layer increased from 53.7 μm to 66.3 μm . Furthermore, the rare earth composite boride layer demonstrated a hardness ranging from 1200-1700 HV, with a gentle hardness gradient. In the adhesion test, there was only slight detachment of the rare earth boride layer, suggesting that rare earth elements effectively reduce the brittleness of the boride layer and enhance its interface adhesion.

Keywords: AISI H13 Steel, composite boronizing, microhardness, adhesion.

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1. Introduction

AISI H13 steel is extensively utilized in the production of different forging dies, hot extrusion dies, and die-casting dies owing to its exceptional hardenability, red hardness, and thermal fatigue resistance [1, 2]. However, it often fails due to thermal erosion, friction and corrosion during metal forming and processing, which greatly reduces its service life [3]. Consequently, it is necessary to use thermochemical treatment technology to improve its surface properties and extend its service life.

Solid boronizing is a highly effective surface treatment method that falls under the category of high-temperature chemical heat treatments [2, 4, 5]. It involves a chemical reaction between the boron source and the activator at elevated temperatures, resulting in the formation of a boride layer on the metal surface with unique physical and chemical properties [6, 7]. The boride layer offers several advantages over traditional surface treatment methods such as carburizing, nitriding, and carbonitriding. These advantages include high hardness, exceptional wear resistance, resistance to high temperature oxidation, thermal fatigue resistance, and corrosion resistance [8-10]. Consequently, solid boronizing finds extensive applications in the surface treatment of various alloys, including steel, nickel, and aluminum.

Zhang [11] prepared boride layers with high hardness and self-lubricating properties on Al-CoCrFeNi2.1 surface by powder embedded boride infiltration method. Guryev [12] obtained a boride coating with a thickness of 80-100 μm on the surface of VT1-0 titanium, which exhibited high hardness. Chen [13] utilized the silicon-free powder filling and electrochemical boronizing method to create a boride layer on CoCrNiMEA.

However, boride coatings are known for their high brittleness, which limits their application range. The researchers discovered that rare earth atoms have low electronegativity and strong reducibility, resulting in strong chemical activity.

This chemical activity promotes the diffusion of B atoms in the metal during boriding treatment, leading to grain refinement of the columnar Fe_2B phase and purification of crystal grains. As a result, the brittleness of the boride layer is effectively reduced, and its performance is improved [14, 15]. Among the rare earth elements, Ce is the most abundant, and rare earth chlorides are commonly found compounds that are easy to prepare and relatively inexpensive.

This paper discusses the co-infiltration of boron and rare earth into AISI H13 steel using the paste boronizing method. It focuses on the impact of rare earth on the microstructure, hardness, and adhesion of the infiltration layer on the surface of AISI H13 steel.

2. Experimental materials and methods

2.1. Experimental materials

In this paper, a quenched and tempered AISI H13 steel with a chemical composition of 0.32-0.45 % C, 0.81-1.20 % Si, 0.20-0.50 % Mn, 4.75-5.50 % Cr, 1.10-1.30 % Mo, 0.80-1.20 % V and a balance of Fe, and a hardness of 500 ± 20 HV, was used. The specimens were machined to a size of $20 \times 15 \times 10$ mm³ using EDM wire cutting method. The surface of the specimens was polished with 180-1000 grit sandpaper and ultrasonically cleaned in alcohol for 10 min and blown dry for use.

The boronizing paste used in this experiment was formulated by the laboratory and consisted primarily of KBF_4 , B_4C , C, and CeCl_3 . A specific proportion of the reagent was mixed with deionized water and evenly applied onto the H13 steel, resulting in a paste thickness of approximately 5 mm. Subsequently, the samples were dried in a drying oven at 200 °C for 2 hours. Following the drying process, the samples were placed in an SX2-4-17TP resistance box for heating at a temperature of 950 °C for a duration of 4 hours, and

the sample is heated up with the furnace cooled to room temperature.

2.2. Experimental methods

After the boronizing process, the sample was removed from the furnace and the surface paste was gently crushed. It was then soaked in deionized water for 15 minutes, followed by ultrasonic cleaning in alcohol for another 15 minutes. After drying, the sample was cut using a wire electric discharge machine and placed in an inlay machine (YZXQ-2) for inlaying. Once the inlay was completed, it was transferred to an automatic polishing machine (YMPZ-1) for rough grinding, fine grinding, and polishing. The rough and fine grinding stages involved using water abrasive paper ranging from 180 to 2000 grain size, while the polishing cloth was made of flannelette and the polishing agent used was 2.0, 1.0, and 0.5 μm diamond spray polish. Finally, the sample was corroded with a 6% nitric acid alcohol solution for 3-5 seconds. The microstructure was studied using a scanning electron microscope (SEM, TESCAN MIRA LMS), and the thickness of the boride layer was measured using the measurement tool provided with an optical microscope (OM, OLYMPUS-DSX-HRUF). Five representative peaks and five troughs were selected and their average was calculated. The microhardness of the cross-section of the boride layer was measured using a Vickers hardness tester (HV-1000) under a load of 50 g and a loading time of 10 s. Adhesion was tested by the VDI 3198 standard with indentation of Rockwell hardness (HRC, HR-150A) at a load of 150 kg [16].

3. Results and discussion

3.1. Microstructure

Figure 1a, b shows the SEM images of the cross-sectional morphology of the boride layers of H13 steel samples with the added rare earth $CeCl_3$ content of 0.4 %, respectively. It can be seen that the cross-sectional microstructure of both sets of boronized samples consists of three layers: the boride layer, the transition layer and the matrix, with the boride layer showing a classical sawtooth shape. A large number of pores were observed in the single boronizing (Fig.1a). Pores are primarily formed during the growth process of the boride layer. Initially, the supersaturated lattice vacancies on the surface come together to create pore nuclei, and the boronizing atmosphere enters these nuclei through the grain boundaries of the primary borides. This leads to increased stress and the formation of solid infiltration, resulting in the formation of pores within the boron layer [17]. After the addition of 4 % $CeCl_3$, a more denser structure of the boride layer becomes apparent, with only a small number of pores observed. This suggests that rare earths can effectively enhance the growth of the boride layer. It was also observed that the thickness of the transition layer of the sample increased after the addition of rare earths. The transition layer is mainly composed of compounds of C and Si that are insoluble in borides, and under the catalysing effect of rare earths, more boron atoms diffuse downward, and C and Si atoms will also diffuse downward more, resulting in an increase in the thickness of the transition layer.

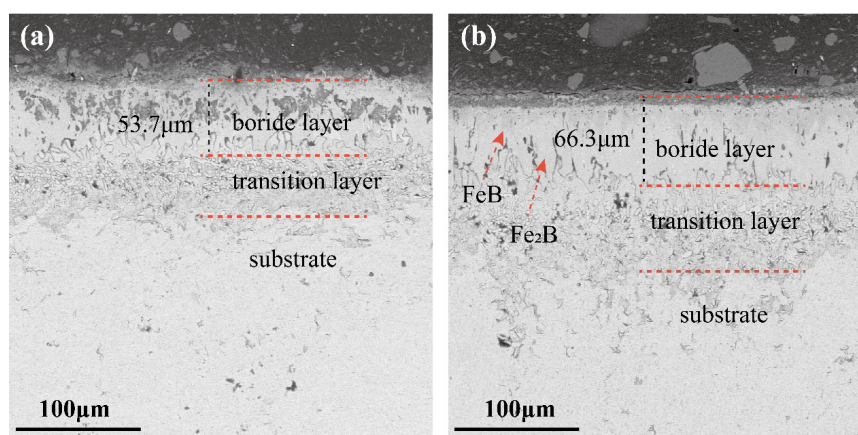


Fig.1. Cross-section microstructures of sample boronized at 950 °C for 4 h: (a) single boronizing; (b) $CeCl_3$ -composite boronizing

Рис.1. Микроструктуры поперечного сечения образца, борированного при 950 °C в течение 4 ч: (а) однократное борирование; (б) $CeCl_3$ -композитное борирование

The thicknesses of the single and rare earth boride layers are 53.7 and 66.3 μm , respectively.

The addition of $CeCl_3$ rare earth significantly increases the thickness of the boride layer. This can

be attributed to two main reasons. Firstly, rare earth elements have lower electronegativity, leading to the generation of more active boron atoms during the boronizing process. Additionally, rare earth elements possess strong reducibility, which helps remove the oxide film on the workpiece surface and allows for better adsorption of active boron atoms and rare earth elements on the sample surface. Secondly, the atomic radius of rare earth elements is approximately 40 % larger than that of iron atoms. This results in lattice distortion around the iron atoms, creating more vacancies and dislocations in the lattice. As a result, the diffusion activation energy decreases and diffusion channels increase, facilitating the accelerated diffusion of boron atoms into the substrate [2, 18]. Consequently, the boride layer thickness shows a significant increase upon the addition of 4 % CeCl_3 .

3.2. Microhardness

Figure 2 presents the microhardness gradient map of the boride layer on the AISI H13 steel sample. The microhardness range of the boronized samples is observed to be between 1200-1700 HV, significantly higher than that of the substrate (500 HV). The sub-surface of the boride layer exhibits the highest hardness value due to the presence of defects such as porosity and pores in the surface layer. Furthermore, due to the high number of pores in the single boride layer, it can be seen that its hardness is more variable and lower in the middle. Notably, the microhardness gradient of the rare earth boride layer is more moderate compared to that of the single boronized sample. The diffusion of C and Si compounds into the transition layer region during the boronizing process leads to lower hardness in the transition zone of both the single boronized and rare earth boronized samples, as compared to the substrate.

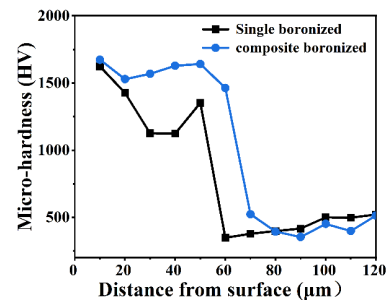


Fig.2. Cross-section hardness profile from the surface to the substrate of single and composite boride layers

Рис.2. Профиль твердости поперечного сечения от поверхности до подложки одиночных и составных слоев бориды

3.3. Adhesion

Figure 3 illustrates the Rockwell hardness indentation morphology of single and composite boride layers. The figure demonstrates that when subjected to a load of 150 kg, the single boride layer completely detaches (Fig.3a), whereas the rare earth composite boride layer only partially detaches (Fig.3b). This observation suggests that rare earth is effective in reducing the brittleness of the boride layer and improving its interfacial adhesion. The adhesion and brittleness of the boride layer are primarily influenced by the Fe_2B and FeB phases. The brittleness of Fe_2B and FeB arises from the uneven distribution of spatial bonds and the weak bond energy of the B-B atomic bonds along the [002] direction. The introduction of rare earth CeCl_3 enhances the boronizing reaction rate, diffusion rate, and nucleation rate of boron atoms, while also strengthening the bond energy of B-B atoms. Consequently, this leads to improved growth of the boride layer and a reduction in internal stress [19]. Therefore, the rare earth composite boride layer exhibits favorable interfacial adhesion.

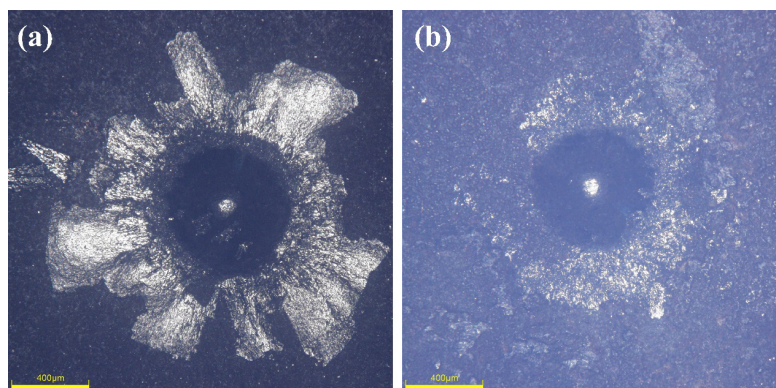


Fig.3. Rockwell indentation morphology of single and composite boronized layers: (a) single boronizing; (b) CeCl_3 -composite boronizing

Рис.3. Морфология вдавливания по Роквеллу одиночных и составных борированных слоев: (а) одиночное борирование; (б) CeCl_3 -композит борирование

4. Conclusions

In this study, for AISI H13 steel, a dense sawtooth-like boride layer morphology was obtained by heating at 950 °C for 4 h using a paste rare earth composite boronizing method, and the boride layer was essentially free of pores and cracks. The thickness of the boride layer increased to 66.3 μm with the catalytic effect of 4 % rare earth CeCl₃. The hardness of the rare earth boride layer ranged from 1200-1700 HV, with a gradual decrease in hardness gradient. Additionally, the inclusion of rare earth CeCl₃ enhanced the boronizing reaction rate, diffusion rate of boron atoms, and nucleation rate. This led to a reduction in internal stress within the boride layer and improved interfacial adhesion of the rare earth composite boride layer.

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