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ИССЛЕДОВАНИЕ ВЛИЯНИЯ БОРИРОВАНИЯ С РЕДКОЗЕМЕЛЬНЫМИ ЭЛЕМЕНТАМИ НА СВОЙСТВА ШТАМПОВОЙ СТАЛИ Н13

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Аннотация. В данной работе проведено борирование редкоземельными элементами на подшипниковых ковочных штампах с целью изучения влияния редкоземельных элементов на структуру и свойства борирующего слоя, анализа и сравнения микроструктуры слоя, микротвердости, хрупкости, жаростойкости и износостойкости исходного слоя образца, одиночное борирование и редкоземельное борирование образцов. Результаты показывают, что после борирования редкоземельными элементами структура ковочного штампа подшипника становится более компактной и однородной. Наибольшая твердость достигла 1612,69 HV (на расстоянии 60 мкм от поверхности борирующего слоя). В 60-минутном испытании на трение и износ потеря веса образца, борированного с редкоземельными элементами составила всего 1/4 от исходного образца и 2/5 от потери веса образца после борированием, в то время как коэффициент трения поддерживается на уровне около 0,23. При высокой температуре 800 °C окислительное увеличение массы при борировании с редкоземельными элементами составило всего 68 % от исходного образца. Можно сделать вывод, что процесс борирования с редкоземельными элементами может улучшить структуру борирующего слоя, стойкость к высокотемпературному окислению и износостойкость поверхности ковочного штампа подшипника, а также уменьшить ее хрупкость.

Ключевые слова: штамповая сталь Н13, редкоземельные элементы, борирование, микротвердость, стойкость к высокотемпературному окислению, износостойкость.

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Original article

INVESTIGATION OF THE INFLUENCE OF BORIDING WITH RARE EARTH ELEMENTS ON THE PROPERTIES OF H13 DIE STEEL

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Abstract. In this paper, rare earth boronizing is carried out on bearing forging dies to study the effect of rare earth on the structure and properties of boronizing layer, analyze and compare the layer microstructure, microhardness, brittleness, high temperature oxidation resistance and wear resistance of original sample, single boronizing and rare earth boronizing samples. The results show that after rare earth boronizing, the structure of the bearing forging die becomes more compact and uniform. The highest hardness is up to 1612.69 HV (at the position of 60 μm from the boronizing layer surface). The brittleness is reduced. In the 60min friction and wear test, the weight loss of the rare earth boronizing sample is only 1/4 of the original sample, and 2/5 of the weight loss of the single boronizing sample, while the friction coefficient is maintained at about 0.23. At a high temperature of 800 °C, the oxidative weight gain of rare earth boronizing is only 68 % of the original sample. It can be concluded that the rare earth boronizing process can improve the structure of boronizing layer, the high temperature oxidation resistance and wear resistance of the bearing forging die surface, and reduce its brittleness.

Keywords: H13 die steel, rare earth elements, boronizing, microhardness, high temperature oxidation resistance,wear resistance.

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Introduction

The failure of metal parts usually comes from the surface or sub-surface of the workpiece [1], which is mainly because the surface hardness and wear resistance of the parts do not meet the service requirements [2-4]. Surface modification can improve the surface properties of metal materials (such as wear resistance, hardness, corrosion resistance and oxidation resistance) [5-7]. Boronizing, as a thermochemical surface strengthening method, is one of the effective ways to improve the surface properties of workpiece [8-10]. Compared with other conventional surface treatments, this technology is characterized by simple process, low cost and no pollution [11, 12], which can significantly improve the wear resistance and hardness of the workpiece, thus improving the service life of the workpiece and saving production costs for enterprises [13-15].

H13 steel is one of the main materials most commonly used in bearing forging dies at present [16, 17]. Li et al. conducted low-temperature boronizing, high-temperature boronizing, vacuum quenching and tempering on H13 steel respectively, and the results show that: The weight loss and weight loss rate of unboronizing, low-temperature boronizing and high-temperature boronizing samples decrease successively [18]. The anti-loss performance of high-temperature boronizing samples is better than that of low-temperature boronizing samples, and the hardness of boronizing layer is about 1500 HV. Adding rare earth elements during boronizing can promote the diffusion of boron atoms, which is a valuable research direction and there are also a few studies at home and abroad [19]. Peng et al. added CeO_2 rare earth boronization to Ti_6Al_4V alloy, which not only improved the friction coefficient, but also made the alloy have better corrosion resistance [20]. Zhu et al. studied the boride growth kinetics of TC21-DT alloy after rare earth boronizing, and the experimental results showed that without adding rare earth, the activation energy of TC21-DT alloy is reduced to 58.13 KJ/mol, about 40 % lower than that of conventional boronizing layer [21]. Wang et al. conducted rare earth boronizing on 45 steel, and found that the wear resistance of the permeable layer increased by 1.25 times after compound [22].

Compared with the performance of rare earth boronizing layer obtained by single boronizing, adding rare earth elements into boronizing agent can improve the diffusion rate, further improve the performance of boronizing layer, and prolong the service life. Therefore, this paper will add rare earth elements into the paste to study the structure and performance of boronizing layer of H13 bearing forging die after adding rare earth elements.

Materials and methods

Materials and process flow

The material used for the experiments is annealed H13 bearing forgings (microhardness 450-580 HV), which are machined into 20 mm × 10 mm × 10 mm specimens by EDM wire cutting (Fig.1). The surface of the specimen is smoothed and polished using different types of sandpaper. The paste boronizing method is adopted, and the reagents mainly include KBF_4, B_4C, CeO_2 and so on.

Fig.1. Bearing forging die

Рис.1. Штамповка подшипников

The sample process flow is as follows: mechanical grinding and rust removal → cleaning and oil removal → paste preparation → paste coating (thickness of about 3-5 mm) → drying preheating (150 °C insulation 2 h) → high temperature boron penetration (850 ~ 950 °C insulation 3-5 h) → air cooling → microstructure observation and performance testing.

The friction and wear test are carried out at room temperature on the MMS-2A-2 friction and wear tester. The frictional wear test conditions are: dry friction, no added lubricant, vacuum hardened 55 steel selected for the frictional substrate, experimental force of 50 N, no rotation of the upper specimen shaft and 200 r/min speed of the lower specimen shaft. After each experiment, the sample surface is cleaned with acetone and weighed on a PTT-A+200 electronic balance.

Experiment

The weight increase method is selected for the high temperature oxidation resistance experiments and the air is connected to the SA2-1-17TP chamber atmosphere furnace for the experiments. It is carried out at 800 °C. Before starting the experiment, the three groups of specimens are gently polished with metallographic sandpaper for corners and burrs, then the dimensions are measured and the surface area is calculated, and the specimens is washed and blown dry with acetone, dried in a vacuum drying dish for one hour and weighed, then placed in a crucible baked at 50 °C. When the temperature rose to working temperature, the specimens and crucible are simultaneously placed in the SA2-1-17TP chamber atmosphere furnace for high temperature anti-oxidation experiments. After each experiment the sample is weighed on an electronic balance PTT-A+200.

The formula for the oxidation weight gain value is shown in equation (1).

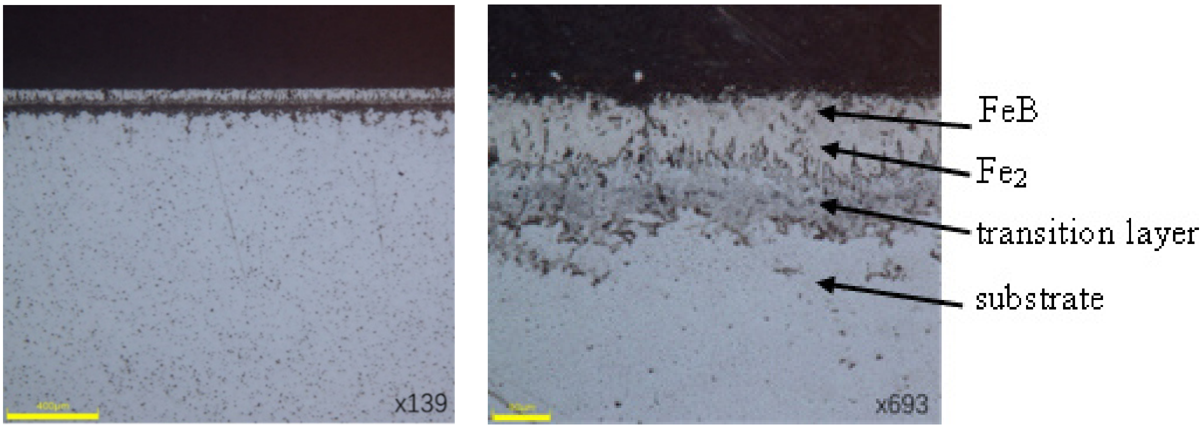
$$W^{+} = \frac{G - G_0}{S}. \quad (1)$$

Where: W^{+} is oxidation weight gain value (mg/cm^2), G_0 is the weight of the specimen and container before the test (mg); G is the weight of the specimen and container after the test (mg); S is the surface area of the specimen (cm^2).

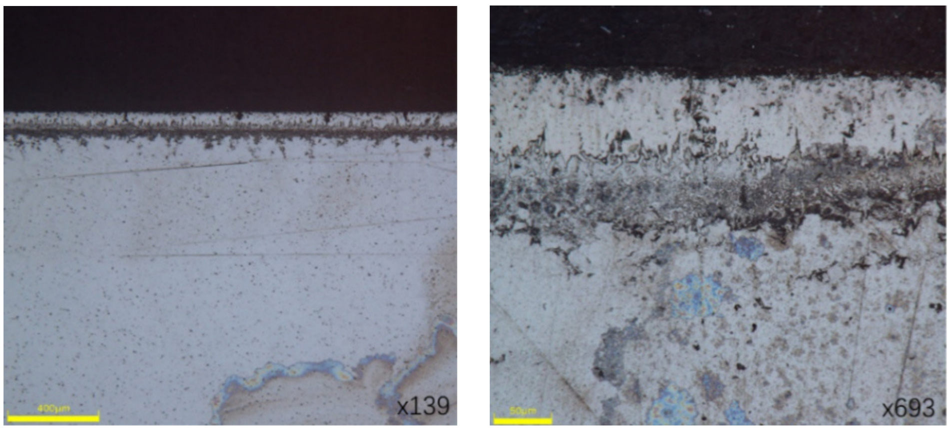
Results and analysis

Microhardness and microstructure of rare earth boronizing layer

Figure 2 shows the microure of boronizing layer of bearing forging die. It can be seen from the Figure that the boronizing layer is FeB, Fe_2B, transition layer and matrix structure from the outside to the inside. Figure 2 (a) shows single boronizing layer, with a large number of holes in the boronizing layer. This is because boron atoms have low activity and slow diffusion, resulting in too low concentration of boron atoms adsorbed on the surface of the material and uneven diffusion of boron atoms inside the material. Therefore, the boronizing layer formed is sparse and has many holes. As shown in Figure 2 (b), after adding rare earth element zigzag structure of boronizing layer presents classic, dense and uniform, combining boronizing layer and matrix, no holes and cracks occurred, good surface quality, but the boronizing layer organization of zigzag sharp enough, this is because the H13 steel for alloy steel, high content of alloy element, in the process of high temperature boronizing, alloying elements is in the transition layer. The tip of the boronizing layer is weakened. Therefore, the addition of rare earth elements can improve the activity of boron atoms and promote the diffusion of boron atoms.



a)



b)

Fig.2. Structure diagram of boronizing layer: (a) single boronizing (950 °C, 4 h); (b) bronizing with rare earth (950 °C, 4 h, 4 % CeO_2)

Рис.2. Структурная схема борирующего слоя: (а) однократное борирование (950 °C, 4 ч); (б) борирование с редкоземельными элементами (950 °C, 4 ч, 4 % CeO_2)

Figure 3 shows the microhardness gradient of the boronizing layer of each sample. It can be seen from the figure that the maximum hardness of the bearing forging die sample treated with rare earth boronizing is 1612.69 HV (at the position of 60 μm from the boronizing layer surface), and that of the bearing forging die sample treated with single boronizing is 1482.16 HV. By comparison, the hardness of bearing forging die through compound boronizing is better than other specimens, because the addition of rare earth element (CeO_2) can improve the activity of boron atoms and promote the diffusion of boron atoms, so as to reduce the hole defects of boronizing layer of bearing forging die sample.

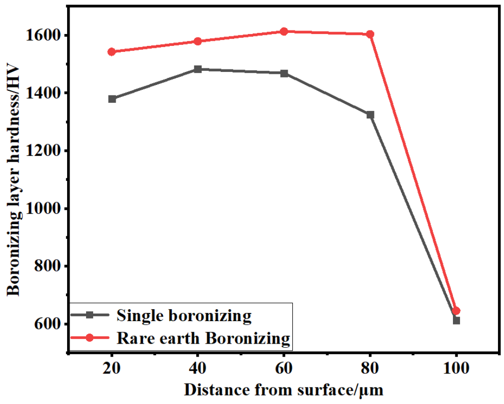


Fig.3. Microhardness of boronizing layer

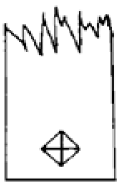
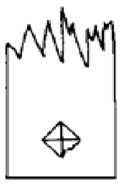
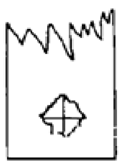
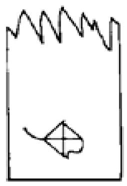
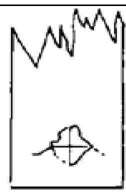
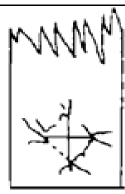
Рис.3. Микротвердость борированного слоя

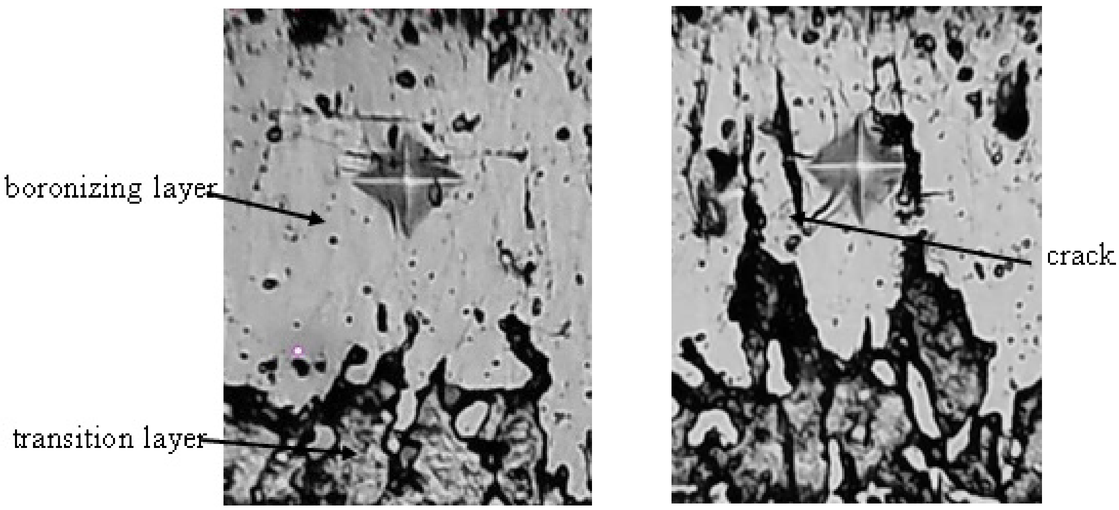
Performance of boronizing layer
Brittleness

The brittleness of the boronizing layer is evaluated by hardness indentation method. The experimental equipment is 1000 HV Vickers microhardness tester. Two groups of samples are selected for the brittleness experiment, which are rare earth boronizing sample and single boronizing sample respectively. The static indentation test is carried out by applying 200 g force 30 μm away from the boronizing layer surface. The brittleness rating is carried out on the size and shape of the indentation, and the brittleness evaluation table is shown in Table 1 [23]. As can be seen from Figure 4 and Table 1, the brittleness level of rare earth boronizing is 2, and that of single boronizing is 4, and the boronizing layer with rare earth element added has low brittleness. This is because the boronizing layer is mainly composed of FeB and Fe_2B in single boronizing treatment. Due to the existence of the inherent brittleness of FeB and Fe_2B, brittle fracture of the boronizing layer is caused when a large load is applied. The brittleness of boronizing layer is low, because the added rare earth elements exist in the boronizing layer in the way of solid solution, which changes the valence electron structure of the boronizing layer, improves the inhomogeneity of spatial bond, strengthens the B-B bond of the boronizing layer, and inhibits the generation of cracks.

Table 1. Brittleness rating

Таблица 1. Оценка хрупкости

Brittleness level	1	2	3	4	5	6
Indentation diagram						



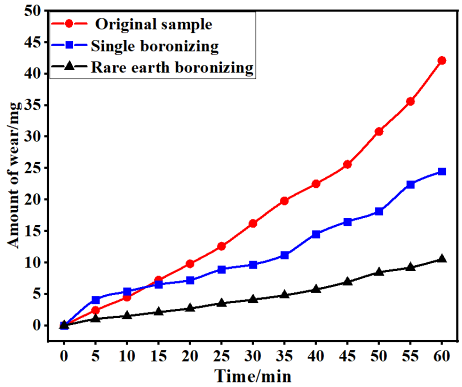
а) б)

Fig.4. Structure morphology: (a) rare earth boronizing; (b) single boronizing

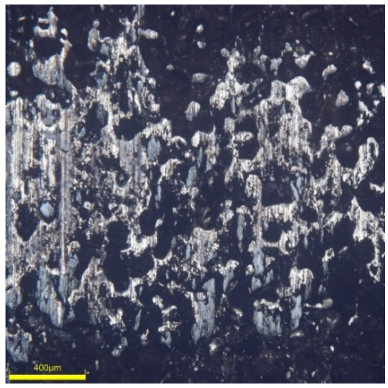
Рис.4. Морфология структуры: (а) редкоземельное борирование; (б) однократное борирование

Wear resistance

Wear resistance is a very important index of bearing forging die. The original sample, single boronizing and rare earth boronizing samples were selected for friction and wear tests. During the experiment, the sample was removed from the wear tester every 5 minutes to measure the quality. Figure 5 (a) shows the relation curve between friction weight loss and wear time of bearing forging die. It can be seen from the Figure that the weight loss of the original sample increases linearly with the increase of time, that is, the longer the time of wear, the more weight loss.



a)



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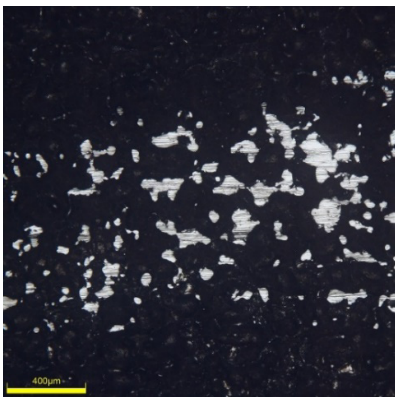

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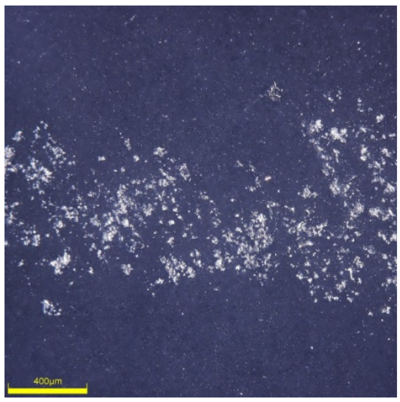
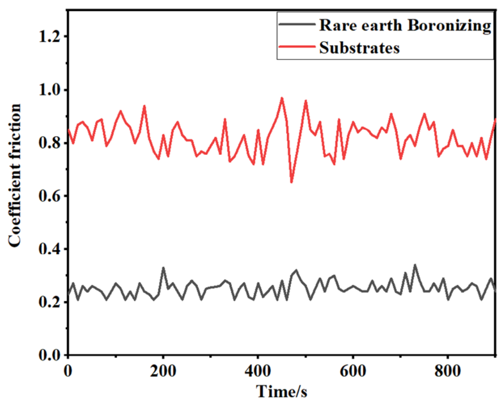


d)



e)

Fig.5. Friction performance of bearing forging die: (a) relationship between wear and wear time; (b) wear scar morphology of untreated bearing forging die; (c) wear scar morphology of ordinary boronizing sample of bearing forging die; (d) wear scar morphology of rare earth boronizing sample of bearing forging die; (e) friction coefficient

Рис.5. Характеристики трения ковочного штампа подшипника: (а) взаимосвязь между износом и временем износа; (б) морфология рубца износа необработанного штампа для поковки подшипника; (в) морфология рубца износа обычного борированного образца штампа для поковки подшипника; (г) морфология рубца износа образца борированного редкоземельного элемента штампа для поковки подшипника; д) коэффициент трения

The weight loss of single boronizing sample was large in the early stage, and gradually stabilized after 10 minutes. Because there are many pores in the sample layer after single boronizing, the roughness is large. During the operation of the friction pair, the roughness is slowly ground down, and the wear trend begins to slow down. In the 60 min friction test, the weight loss of the rare earth boronizing sample is only 1/4 of the original sample, and 2/5 of the weight loss of the single boronizing sample. If the wear resistance of the three groups of samples is measured by weight loss, the boron wear resistance of the three groups of samples from good to bad is: rare earth boronizing sample, single boronizing sample, original sample.

Figure 5 (b), (c) and (d) are the morphologies of wear marks of the three groups of samples after friction for 15 min at a load of 100 N. In Figure 5 (b), the wear marks show furrow characteristics, and the boronizing layer in the wear marks has local peeling and crack characteristics. Compared with Figure 5 (b), the wear marks in Figure 5 (c) and Figure 5 (d) are shallow and narrow, and the surface wear is lighter. After boronizing the bearing forging die, the width of the wear mark of the bearing forging die is obviously narrower than that of the bearing forging die, and the morphology of the wear mark changes greatly. The wear mark is shallow and smooth, and there is no penetration mark on the surface of the boronizing layer. After the rare earth boronizing treatment, the width of the wear mark of the rare earth boronizing sample becomes narrower obviously, and the morphology of the wear mark also changes greatly. This is because under the rare earth boronizing process conditions, the boron atom activity is enhanced, the boronizing layer and the matrix are more closely combined, and the fatigue wear is mainly on the whole.

It can be seen from Figure 5 (e) that the friction coefficient of bearing forging die matrix material is much larger than that of rare earth boronizing sample. In the initial stage, the friction coefficient of the matrix sample fluctuates around 0.85 with the increase of the friction test time, and reaches the maximum value of 0.96 at about 450 s. Then, the friction coefficient gradually stabilizes, and the average friction coefficient is 0.86. The friction curve of rare earth boronizing sample fluctuates in a small range around 0.24, and the friction coefficient does not increase sharply.

High temperature oxidation resistance

Due to the high working temperature of the bearing forging die (500-800 °C) [24], the high temperature oxidation resistance test of bearing forging die is carried out for the original sample, single boronizing sample and rare earth boronizing sample at 800 °C × 8 h. In the experiment, the samples were taken out and cooled to room temperature every 1h before weighing, and the oxidation weight gain was calculated. Figure 5 shows the change curve of H13 steel's oxidation weight gain at high temperature at 800 °C. As shown in the Figure, the oxidation weight gain of the three groups of samples increases rapidly at first and then slows down as time goes by. This is because the oxide film is formed on the surface of the samples as time goes by, which can inhibit the further oxidation of metal. After 8 hours of oxidation test, the oxidative weight gain of rare earth boronizing is 68 % of the original sample, which is 95 % of that of single boronizing, which means that at high temperature, there is little difference in weight gain between the rare earth boronizing layer and the single boronizing layer, indicating that the addition of rare earth has little effect on the high temperature oxidation resistance of the boronizing layer at high temperature.

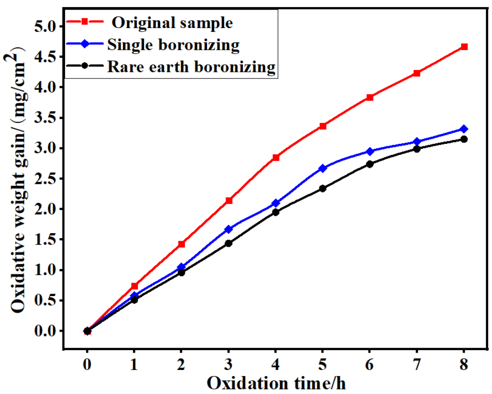


Fig.6. High temperature oxidation weight gain curve at 800 °C

Рис.6. Кривая увеличения веса при высокотемпературном окислении при 800 °C

Conclusions

In this paper, the microstructure and properties of the rare earth boronizing layer of the bearing forging die are studied. Through this method, the wear resistance and high temperature oxidation resistance and other properties of the bearing forging die are greatly improved. The results show:

(1) CeO_2 can improve the microstructure of the boronizing layer and make the boronizing layer more compact and uniform, thus improving the hardness of the boronizing layer. When 4 % CeO_2 is added, the microhardness of bearing forging die is 1612.69 HV (at the position of 60μm from the

surface), which is about 3 times higher than that of original sample.

(2) Rare earth boronizing significantly reduces the brittleness of the boronizing layer, because the added rare earth elements exist in the boronizing layer in the way of solid solution, which changes the valence electron structure of the boronizing layer.

(3) After rare earth boronizing, the surface friction performance of the bearing forging die has been greatly improved. The weight loss of the rare earth boronizing bearing forging die is only 1/4 of the original sample, and 2/5 of the weight loss of the single boronizing sample. The width of the wear scar is obviously narrowed, only local spalling phenomenon exists, and the friction coefficient is also reduced to 0.23. The high temperature oxidation resistance of the bearing forging die at 800 °C is improved compared with the original sample.

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