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Microstructure and wear properties of niobium carbide particulates gradient-distribution composite layer fabricated in situ

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ABSTRACT

Niobium carbide (NbC) particulates gradient-distribution composite layer was successfully synthesized by in situ techniques. The formation mechanism, microstructure evolution and wear properties of the composite layer were investigated in detail. The results show that the thickness of the composite layer is about 1360 μm after the heat treatment at 1175 °C for 1 h. From the surface to the matrix, the volume fraction of NbC particulates continuously decreases while the average particulate diameter gradually increases from 213 nm to 1.46 μm. Correspondingly, the micro-hardness and relative wear resistance almost gradually decrease from the top surface to the matrix.

In order to discuss the wear mechanism clearly, the composite layer was artificially divided into four regions according to the continuously variation of composition and microstructure. The wear mechanism of NbC particulates high-density zone (region A) is mainly characterized by the intergranular micro-cracks and micro-ploughing. The relative wear resistance of this region is 75 times higher than that of HT300. The wear mechanism of NbC particulates partly gathering zone (region B) is micro-ploughing and part of micro-cutting. NbC particulates dispersing zone (region C) is slight micro-cutting and a small amount broken NbC particulates. And near-matrix zone (region D) is severe micro-cutting with broken and re-embeding NbC particulates in the matrix. And the wear resistance of the four zones is higher than that of matrix.

1. Introduction

Carbides of transition metals in No. 5 (V, Nb, Ta) and 6 (Cr, Mo, W) groups of the periodic table exhibit high hardness, good wear resistance, excellent corrosion resistance and other advantages [1]. As one of the refractory carbides, niobium carbide (NbC) presents high hardness (19.60 GPa), high melting temperature (3600 °C), large thermal expansion coefficient and high temperature deformation resistance [2,3], comparable to VC, TaC, Cr_7C_3, etc [4–8]. Furthermore, NbC also has a much lower density (7.79 g/cm^3) and stronger antioxidant capacity compared to common reinforcing ceramic WC. Therefore, it would be a very suitable material used as reinforcing phase in composite layer, which is responsible for the resistance to wear, thermal loads and corrosion.

As well known, surface treatments are using hard coatings or reinforcing phase to protect the substrate. They have been paid more and more attention to by the researchers, because the damage and failure occur mainly on the surface of components and tools. The surface reinforcing layers can increase tool life or efficiency powerfully in industrial machinery. NbC using as reinforcing phase in surface layer have attracted a lot of interesting due to its rare combination of valuable properties. At present, there are various technologies [9–19] for fabricating NbC reinforcing layer. Such as niobium coatings with highest thickness of 25.3 nm were deposited on graphite by magnetron sputtering [12]. Sen [13,14] studied the sliding wear behavior of the NbC coatings prepared by TRD method on AISI 1040 steel, whose results suggested that the friction coefficients and the wear rate of the coated steel were lower than those of the uncoated steel. But the highest thickness of NbC layer fabricated by them is only 12 ± 2 μm. Furthermore, Oliveira et al. [15] proved that the micro-abrasive wear resistance of the NbC layers by PVD was considerably superior to uncoated AISI H13. Shiri [9] fabricated Cu/NbC functionally graded structure by PM. And the sample showed high wear resistance and hardness on the composite surface.

However, most of these layers were conventional homogeneous composite materials [9–11], which yield the benefits of

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the reinforcing phase NbC but without any transition at the interface. In addition, most of the NbC layers obtained by them are thin. These cases would result in stress concentration at the interface between the reinforcing layer and matrix. Therefore, it is important to put forward surface gradient-distribution reinforced layer with a comparative thickness, which can provide a number of distinct advantages [9]. The gradually varied interface leads to little stress concentration and similar thermal expansion coefficients between the layer and the matrix, and consequently improves the bond strength. On the other side, a majority of these methods [10–19] for fabricating NbC layers not only require complex equipments and high reaction temperatures, but also lead to impurity of products, weak bond strength and extra energy consumption. However, in situ synthesis mentioned in this work is a promising technology [20–22] because of the following reasons: (1) controllable thickness of reinforcing layer; (2) controllable reinforcing phase in type, size, and distribution; (3) perfect metallurgical combination of the interface between the particulates and matrix; (4) simple equipments and low cost.

In this study, we fabricated NbC particulates gradient-distribution reinforced gray cast iron matrix composite by in situ synthesis. Correspondingly, the formation mechanism of composite was investigated. In addition, the Vickers micro-hardness and two-body abrasion tests were carried out from the top surface to the matrix. By combing the SEM observation of worn surface and the wear properties of different regions, the individual wear resistance mechanism was analyzed and compared.

2. Experimental procedure

For the in situ fabrication of carbide particulates gradient-distribution composite, HT300 and niobium plate were used as source materials, which provide the carbon and niobium atoms during the reaction, respectively. The chemical compositions of the gray cast iron and the niobium plate are listed in Table 1. Firstly, we prepared Nb/Fe composite prefabricated body by casting. Namely the melted gray cast iron was poured into a graphite crucible which was placed with a niobium plate at the bottom. Then, the prefabricated body was immediately covered with quartz sand to avoid crack generation and cooled down to room temperature. Secondly, the body was put into at another graphite crucible and heat-treated at 1175 °C for 1 h in a horizontal tube furnace (Suzhou Nutac Electro Mechanic Co. Ltd., China) with a modest flow of argon gas (5 ml/min). After that, the specimen was quenched in water.

After being polished with diamond paste and etched with 4% Nital solution, the microstructures of the specimens were examined by using a JSM-6700 scanning electron microscope (JEOL, Japan). The X-ray diffraction (XRD) data were recorded on a XRD-7000 X-ray diffractometer (Shimadzu, Japan) with CuKα radiation at 40 kV and 40 mA in the 2θ range of 15 – 85°. The micro-hardness of the specimens was measured by a HDX-1000 digital micro-hardness tester consisting of a square-based pyramidal diamond indenter with a 136° angle between two opposite faces. The static load was 50 g, and the dwell time of loading was 15 s. The average micro-hardness was taken from at least three different measurements.

Two-body abrasion tests were performed on a ml-100 pin-on-disk tribometer with alumina abrasives. The composites were cut to pins (Φ6 mm × 15 mm) vertical in the specimen surface. The disk was attached with 320-grit alumina abrasive paper on the surface and rotated with a speed of 60 rpm. The pin specimen was moved against the abrasive paper in a spiral form to ensure fresh supply of abrasive particulates under a normal load of 5 N. The worn alumina abrasive paper was replaced after every 20 m sliding distance. The abrasive wear tests were carried out continuously from the top surface to the iron matrix for the cylindrical pin composite. The weight of worn specimen before and after one circle was measured on a CP224S analytical balance with an accuracy of 0.0001 g. The average weight loss was taken at least three times from different measurements. The abrasive wear property of the each region can be evaluated by the relative wear resistance which can be calculated by the following mathematical expression:

$$\beta = \Delta m_r / \Delta m_c \quad (1)$$

Where β is the relative wear resistance, Δm_c is the abrasive weight of the NbC particulates gradient-distribution, and Δm_r is the abrasive weight of the standard sample (HT300) in the same wear condition.

3. Result and discussion

3.1. The phase composition and microstructure

The X-ray diffract gram of the cross section for NbC gradient-distribution reinforced composite is shown in Fig. 1. It verifies that the composite layer consisted of NbC and α-Fe, without any other impurity phases. The peak intensities of NbC at angles 2θ of 34.72°, 40.32°, 58.34°, 69.72°, and 73.31° are correspondence with a cubic crystal structure [13], which is attached to a group space of [Fm-3m(225)].

Fig. 2 shows the micrographs of a cross-section of the NbC particulates gradient-distribution performed by in situ techniques. There are no obvious interfaces on the whole cross-section, which exhibits perfect metallurgical bonds. The thickness of the reaction zone is approximately 1360 μm. From the enlarged micrographs M1 and M2, the reaction zone can be artificially divided into four regions: niobium carbide particulates high-density zone named region A, niobium carbide particulates partly gathering zone named region B, niobium carbide particulates dispersing zone named region C and near-matrix zone named region D. The microstructure of the composite is similar to that of our previous study [23].

To obtain more information about the different reactive zones, scanning electron microscopy was applied to zoom in the four regions, as shown in Fig. 3. Some parameters about ceramic particulates are listed in Table 2. Region A is characterized almost pure tiny sphere-like niobium carbide particulates with volume fraction 88.89%. The average diameter of particulates in this region A is about 213 nm. As can be seen from Fig. 3(b) of region B, it consists of NbC particulates and a little ferrite matrix. The fine NbC ceramic particulates are partly-gathering. The gathering NbC particulates micro layer and the ferrite matrix micro layer arrange

Table 1
The chemical composition of HT300 and niobium plate (wt%).

Materials	Si	Mn	P	S	C	Fe	Ti	Ta	O	Cu	Nb
T300	1.03	1.04	0.046	0.018	2.57	Balance	–	–	–	–	–
Niobium plate	–	–	–	–	0.004	0.004	0.002	0.05	0.015	0.03	Balance