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Effect of brazing temperature on microstructure and mechanical properties of graphite/copper joints

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ABSTRACT

A novel Ni–Cr–P–Cu filler alloy was designed to join graphite and copper at 1173–1253 K for 10 min, and the effects of brazing temperature on the microstructure and mechanical properties of the joints were investigated. The typical microstructure of the joints was as follows: graphite/Cr_3C_2+Cr_7C_3 reaction layer/Ni_3P+Cu-based solid solution+chromium carbide reaction phases/Cu. On increasing the brazing temperature, the thickness of the brazing layer was reduced, whereas the thickness of the reaction layer at the graphite/filler alloy interface was increased. However, the reaction layer was formed discontinuously at the graphite/filler alloy interface while increasing the brazing temperature to 1253 K, at which the crack propagation mode was through the interface of the graphite/filler alloy. The shear strength first increased then decreased with the increase of brazing temperature, and the maximal shear strength of the joint could reach 60 MPa when the brazing temperature was 1223 K.

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1. Introduction

The International Thermonuclear Experimental Reactor (ITER) is in progress under an international collaboration to achieve the application of magnetic fusion as an energy source and the achievement of a practical fusion reactor. One of the most technically challenging components of the ITER machine is the divertor, which includes the cassette body (CB) and the plasma-facing components (PFCs) [1], followed by the demands of plasma-facing materials. CFCs (where CFC is the abbreviation of carbon fiber reinforced carbon matrix composites) are employed widely in ITER as plasma-facing components due to their high thermal conductivity, excellent thermal shock and fatigue resistance [2]. Moreover, copper is chosen as heat sink materials for its excellent electrical and thermal conductivity, which can effectively conduct electricity or dissipate heat of the component in radiation environment [3,4].

With the rapid development of the automotive industry, the requirements of automotive accessory are increasing rapidly. So electric machines have wide application across the whole industries inevitably, in which commutator is one of the key components. However, most commutators in D.C. motors were made of oxygen-free copper or Ag–Cu alloy, and they had to be replaced annually due to the heavy wear between the carbon brushes and commutators [5]. To improve the wear resistance and prolong the working time, graphite is applied as lubricant on the surfaces of copper commutators due to its high lubrication and abrasion resistance. So it can be easily understood that in order to achieve the wide applications of plasma-facing materials and carbon commutators, the joining of copper (or its alloys) to carbon-based materials has raised wide concern [6–8].

It is well known that graphite has a high melting point (3823 K) and cannot form any stable carbide with Cu; hence, melting and direct diffusion bonding methods cannot be adopted in this situation. In recent years, various bonding technologies have been developed to join graphite to metals, such as adhesive joining [8,9], solid-state bonding [10], and brazing [11–13]. Among these joining methods, brazing has received extensive attention due to its simplicity, high joint strength, good repetitiveness, low cost-effectiveness as well as perfect adaptability of joint size and shape. Considering that wettabilities of most metals on graphite are unsatisfied, the strong carbide-forming elements (such as Ti, Zr, Cr, Mn, Si) must be incorporated in the filler alloys to improve their wettabilities in the graphite/copper system [11,12].

In this research, a novel Ni–Cr–P–Cu filler alloy was designed to join graphite and copper. In filler alloys, nickel-based filler alloys have both good corrosion resistance and creep strength [14,15], and the unlimited mutual solubility of Cu and Ni is beneficial to solve the joining problem with copper. The addition of Cr in the filler alloy can improve wettability dramatically since Cr can react with carbon to form chromium carbides [16]. P element in the filler alloy can reduce the melting point of the filler alloy efficiently according to the Ni–P binary phase diagram [17]. Moreover, P can also improve the wettability and spreading ability of the filler alloy [18]. A small amount of Cu was also added in the filler alloy

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because Cu can improve the electrical conductivity of the joints to a certain degree due to its low electrical resistivity.

In this research, $Ni_{68.4}Cr_{12.6}P_9Cu_{10}$ (wt%) filler alloy was used to join graphite and copper at different temperatures for 10 min. The microstructure, interfacial evolution mechanism and shear strength were studied to access the effect of brazing temperature on the microstructure and mechanical properties of the joints.

2. Materials and experimental procedures

The high-strength graphite used in the experiments was obtained from Shanghai Xi Li Carbon Co., Ltd. and the average shear strength of graphite at room temperature is over 65 MPa. The copper used was a commercial material with the composition of 99.99 wt% (General Research Institute of Nonferrous Metals, Beijing, China). The graphite was cut into the samples with the dimension of $20 \times 5 \times 2.5\ mm^3$ for shear strength test and $5 \times 5 \times 2.5\ mm^3$ for microstructure observation, and the dimensions of copper were $3 \times 4 \times 4\ mm^3$. The bonding surfaces were ground on SiC abrasive papers and then polished using 0.5 μm diamond pastes. Prior to assembling, the specimens were degreased and cleaned with acetone in an ultrasonic bath for 30 min. The filler alloy with an average particle size of 20 μm was composed of $Ni_{68.4}Cr_{12.6}P_9Cu_{10}$ (wt%). Differential thermal analysis (DTA, TGA1600) was used to measure the melting point of the filler alloy in Ar atmosphere at a flow rate of 40 ml/min, showing that the solidus temperature of the filler alloy is 1140 K, whereas the liquidus temperature is 1166 K.

Before assembling, a small amount of cellulose nitrate and octylacetate was added into the filler alloy powder for making a brazing paste, and the mount of the filler alloy powder used for each sample was controlled in the same weight (20 mg). After that, the brazing paste was sandwiched between the graphite and copper, and then the assembly was placed in a graphite jig. A pressure of 1×10^4 Pa was exerted on the brazing assembly to make the filler alloy contact closely with the substrates. The brazing process was carried out at different temperatures (1173–1253 K) for 10 min in a vacuum of $(1.0–3.0) \times 10^{-3}$ Pa. At the beginning, the assembly was heated to 623 K at a rate of 15 K/min and held for 30 min to volatilize the binder. Then the temperature was increased to 1163 K at a rate of 30 K/min and held for 20 min, and later increased to brazing temperature at a rate of 15 K/min, isothermally soaked for 10 min. Finally, the brazing samples were cooled down at a rate of 5 K/min to 473 K and then cooled in a furnace without power.

The shear strength of the brazed joint was measured by an Instron-5569 electronic universal testing machine using a specially designed jig, as shown in Fig. 1. The specimens were loaded by the testing machine at a constant speed of 0.5 mm/min. At least five samples were used to determine the strength of joints for each joining condition. To characterize the microstructure of the joints, the cross-sections of the brazed joints were examined by a scanning electron microscope (SEM, FEI QUANTA 200 F) equipped with an energy dispersive spectrometer (EDS). The phases in the joints were also identified using an X-ray diffraction (XRD, Philips X'Pert) method, and the specimens for the XRD test were cut parallel to the joint surface and then ground to expose the filler alloy at the surface of the specimen.

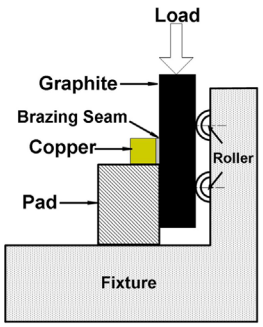

Fig. 1. Schematic drawing of the shear strength test.

3. Results and discussion

3.1. Microstructure characterization of the brazed joints

Fig. 2 shows the morphology and corresponding elements' area distribution images of a graphite/copper joint brazed with Ni–Cr–P–Cu filler alloy at 1223 K for 10 min. A defect-free joint was obtained, which revealed that good wetting and intimate contact were formed between the substrates and filler alloy. The brazing layer can be divided into two regions named zone I and II separately. Zone I is made up of a continuous gray reaction layer close to the graphite with a thickness of 5–6 μm. The central part of the joint (zone II), which has an average thickness of 37 μm, is composed of white phase B, light gray matrix phase C and gray particle phase D.

Fig. 2(b) shows that Cr elements are mainly distributed at the graphite/filler alloy interface, indicating that Cr, as an active element, had diffused towards the graphite and reacted with the graphite at the interface. According to the C–Cr binary phase diagram [16], three kinds of compounds (Cr_3C_2, Cr_7C_3 and $Cr_{23}C_6$) are present, and the free energies of formation for these chromium carbides at 1223 K are all negative [19], suggesting that all the three compounds are thermodynamically favorable. Numerous researches about the nature of chromium carbide phase at the interface have been reported. For instance, Wang et al. [20] introduced the commercial Ni–Cr–P active brazing alloy to join diamond grit to the steel substrate and found that Cr_3C_2 and Cr_7C_3 were formed at the interface between the diamond and filler alloy. Tao et al. [21] studied the wettability and interface thermal resistance of a copper/graphite system with an addition of chromium in the filler alloy, and found that a continuous carbide layer, which consisted of Cr_3C_2 and Cr_7C_3, was formed at the interface zone. Furthermore, Yang [22] investigated the isotherm wetting and spreading behaviors of the molten Cu–Cr alloys with 0.5, 1.0 and 2.0 at% Cr on porous graphite substrates at 1373 K, and claimed that the Cr_3C_2 phase forms preferentially at the Cu–Cr alloy/graphite interface and then the Cr_7C_3 phase. It is worth noting that Cr_3C_2 and Cr_7C_3 are formed at the interface in all of their studies, but until now, it is still hard to fully understand the formation mechanism of the chromium carbide interface layer.

Further analysis of the phases in the joint was carried out using the XRD method. The joint was cut at the graphite side close to the joining interface, and then ground until three positions (illustrated in Fig. 3(a)) were exposed. XRD patterns corresponding to different positions are shown in Fig. 3(b). From the XRD results of graphite/filler alloy interface (plane A), Cr_3C_2 and Cr_7C_3 can be detected. It is well known that the Gibbs free energy of formation of Cr_3C_2 is more negative than the formation of Cr_7C_3 and $Cr_{23}C_6$ in the range of brazing temperature (1173–1253 K). Alternatively, the concentration of carbon is enriched near the graphite, and the critical concentration of Cr for the formation of Cr_3C_2 is smaller than that of Cr_7C_3 and $Cr_{23}C_6$ under the same circumstance [22]; hence, Cr_3C_2 should be formed preferentially at the graphite/filler alloy interface. Then we can safely reach the conclusion that Cr_3C_2 phases are formed preferentially and then the Cr_7C_3 phases at the graphite/ filler interface.

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