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Synthesis of A (Pb_x, Fe_y, C_z) O_e Matrix Composite

Xinjun PEI

Yangjiang Polytechnic, Yangjiang 529566, China

***Corresponding author:** Yangjiang Polytechnic, Yangjiang 529566, China.

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Abstract
A (Pb_x, Fe_y, C_z) O_e (Abbreviated as PFCO) matrix composite bounded with an alumina ceramic was synthesized by mixing the powders of pure lead (Pb), pure iron (Fe), graphite (C) and pure nickel (Ni) with mass percentage of (65%Pb, 25%Fe, 5%Ni, 5%C) under atmospheric pressure at a maximum temperature of 1530℃. And the mixture of pure lead, pure iron, graphite and pure nickel can be used as a new kind of brazing materials for alumina ceramics. The phase composition, cross-sectional microstructure, hardness, ferromagnetism and insulating properties of the PFCO matrix composite were analyzed by means of scanning electron microscope (SEM), energy dispersive spectrum (EDS), optical microscope, Vickers hardness tester, X-ray diffractometer (XRD), a permanent magnet and a multimeter. The PFCO matrix composite exhibits favorable wettability with the alumina ceramic and is bounded to an alumina ceramic through an interface and is composed of the PFCO matrix composite, PbO and Fe3O4. The PFCO matrix composite belongs to ferromagnetism and insulating material with 301Hv.

Keywords: (Pb_x, Fe_y, C_z) O_e Matrix Composite, Alumina Ceramic, Brazing Material, Ferromagnetic Material, Insulating Material.

Introduction

Ceramic materials exhibit a multitude of advantageous properties in physical, chemical and mechanical properties, such as high hardness, high wear resistance, high corrosion resistance, high electric resistance, high thermal stability and chemical stability, that ceramics in the workpieces of ceramics/ceramics and ceramic/metal composite materials have been widely used. The welding between ceramics and ceramics, as well as the welding between ceramics and metals, can be achieved through a variety of methods, including brazing, diffusion welding, self-propagating high-temperature synthesis welding, fusion welding, etc. [1-6]. At present, the metal materials utilized in the preparation of ceramic/ceramic and ceramic/metal composite materials are conductive materials. Ceramic is a good insulating material, if the ceramic and ceramic are welded into composite materials with conductive materials, the welded joints of ceramic and ceramic will inevitably conduct electricity, which will break the overall insulation properties of the ceramic/ceramic composite.

The existing ceramic/ceramic and ceramic/metal welding materials are non-ferromagnetic materials. Ferromagnetic materials have a wide range of applications. If the ferromagnetic materials used as ceramic/ceramic welding materials, this will expand the ceramic/ceramic welding materials. Ferromagnetic materials include iron, nickel and cobalt, etc. Among them, iron is widely used in pesticides, powder metallurgy, hot hydrogen generator, gel propellant, combustion active agent, catalyst, water cleaning adsorbent, sintering active agent, powder metallurgy products, various mechanical parts products, cemented carbide materials products, etc. [7-10]. Compared with iron, lead exhibits the advantages of low melting point, high corrosion resistance and good plasticity. Additionally, X-rays and gamma rays are unable to penetrate it with ease. And it is often processed into plates and pipes, which are widely used in chemical, cable, battery and radiation protection industries [11-13]. Lead and iron are two materials with large differences in physical and chemical properties, and there is a paucity of research literature examining the reaction of these two materials to produce lead-iron alloys. Furthermore, there is no research reports on the use of these two materials as ceramic/ceramic welding materials.

In this study, lead, iron graphite and nickel powders were used as raw materials to prepare a new PFCO matrix composite at 1530℃, and found that the mixture of pure lead, pure iron and pure nickel can be used as a new kind of brazing material for alumina ceramics. The phase composition, microstructure, hardness and ferromagnetism of the product were analyzed by optical microscope, electron scanning microscope (SEM), energy dispersive spectrum (EDS), Vickers hardness tester, X-ray diffractometer (XRD), a permanent magnet and a multimeter.

Experimental Procedure

The raw materials utilized were elemental powders: pure iron powders (99.7% iron mass percentage, particle size 120 mesh), pure lead powders (99.5% lead mass percentage, particle size 100 mesh), pure graphite powder (99.9% pure graphite mass percentage, particle size 300 mesh), and pure nickel powders (99.8% nickel mass percentage, particle size 150 mesh). The four powders were weighed in accordance with their respective mass percentage (65%Pb, 25%Fe, 5%Ni, 5%C). Subsequent to the weighing of the raw materials, the following procedures were carried out: a) Four kinds of powders were put into the ball tank of the ball mill and an appropriate amount of anhydrous ethanol was added to the ball mill, the ball material ratio was 1:1, and the ball milling time was 2 hours; b) The raw material mixture after ball milling was poured into the alumina ceramic crucible and subsequently placed in the drying oven, and dried at 100℃ for 1 hour. After drying, the alumina crucible containing the raw material mixture was placed in a high temperature sintering furnace heated by resistance, heated to 1530℃ at a heating rate of 20℃/m and kept at 1530℃ for 30 minutes, and then cooled with the furnace.

The properties, phase composition and structure of the PFCO matrix composite were analyzed by electron scanning microscope (SEM), energy dispersive spectrum (EDS), optical microscope, Vickers hardness tester, X-ray diffractometer (XRD) and rare earth permanent magnet with a side length of 5mm and a thickness of 0.5mm. SEM was performed to observe the fracture and microstructures of the PFCO matrix composite. The phases present in the PFCO matrix composite were determined using XRD. EDS was used to analyze chemical composition of the PFCO matrix composite. Vickers hardness tester was carried out to test the hardness of the PFCO matrix composite. Rare earth permanent magnet was used to test the ferromagnetism of the PFCO matrix composite. Insulation performance of the PFCO matrix composite was tested by a multimeter.

Results and Discussions

2.1 Bond strength between alumina ceramics and the PFCO matrix composite

The PFCO matrix composite adheres to the alumina ceramic crucible and cannot be removed from the crucible. After breaking the crucible, the crucible breaks into several pieces, and the PFCO matrix composite also breaks into several pieces, but each piece still adheres to the crucible.

The bond strength between alumina ceramics and the PFCO matrix composite has not been tested by special equipment, and no specific value of the bond strength has been obtained. However, when breaking the alumina crucible adhering to the PFCO matrix composite, it is observed that although the PFCO matrix composite is also broken, it remained adhered to the ceramic, which can be considered that the bond strength between the alumina ceramic and the PFCO matrix composite is greater than the general mechanical bond strength. Therefore, the mixture of pure lead, pure iron, graphite and pure nickel can be used as a new kind of brazing material for alumina ceramics.

XRD Analysis

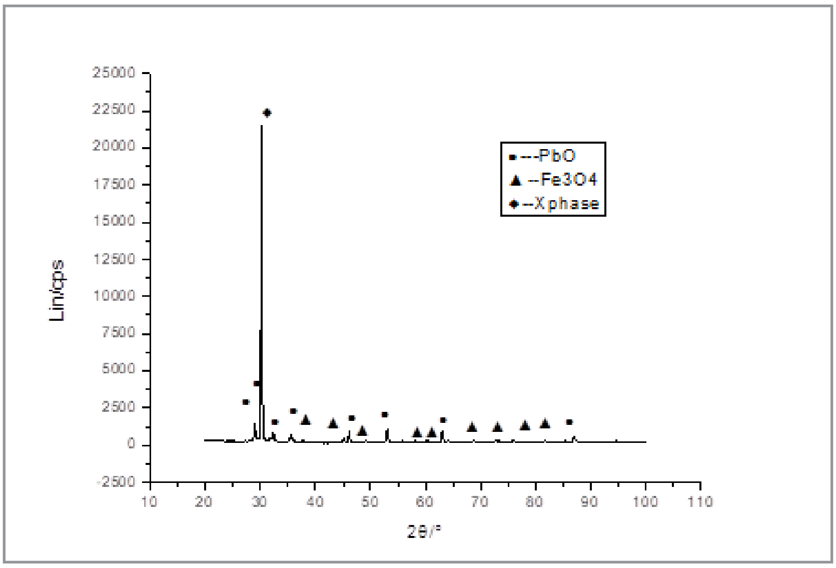


Figure 1 The Results of XRD Analysis

Fig. 1 is the XRD analysis result curve of the PFCO matrix composite. By comparing the peak phase of XRD and the characteristic value of the phases in the special software, it can be seen that the PFCO matrix composite mainly consists of an unknown namely phase-X phase, PbO (lead oxide) and Fe3O4 (ferro tetroxide), among which the X phase content is the largest.

The maximum temperature of the experimental heating process is 1530℃, and this process is carried out in air. During the heating process, the oxygen in the air reacts with Pb and Fe in the raw materials to generate PbO and Fe3O4. At the same time, Pb and Fe may react with each other to generate a new phase. By comparing the phase of the peak in the XRD junction curve with the characteristic values of PbO and Fe3O4 in the special software, it can be seen that some of the phases of these peaks can correspond to the characteristic values of PbO and Fe3O4, respectively, confirming the existing phases PbO and Fe3O4. However, for the strongest main peak, it is difficult to identify a corresponding phase. This may be due to the lack of corresponding phases in the analysis software, and further research is needed to determine the X phase.

Hardness test

A piece of the PFCO matrix composite was selected to make metallographic sample, and then its hardness was tested by Vickers hardness tester. The minimum loading force was used during the hardness testing of the material, and the hardness obtained is 301Hv.

When making metallographic sample, the PFCO matrix composite was polished with metallographic sandpaper, and it is found that ordinary brown corundum sandpaper cannot grind out a smooth plane on the PFCO matrix composite, but the abrasive particles on the metallographic sandpaper were worn off.

The hardness test and metallographic sample preparation process indicate that the hardness of the PFCO matrix composite should be greater than 301Hv. However, due to the brittle and porous nature of the material, it is broken under the pressure of the loading force, preventing the accurate measurement of the true hardness value.

Ferromagnetic Detection

The reference material used to detect ferromagnetism is a piece of 304 austenite steel with a ground surface. In the comparative test, it was found that the 304 austenitic steel cannot be attracted by magnets, while the PFCO matrix composite can be attracted by magnets, even if the magnet was suspended on the PFCO matrix composite, it would not fall.

The magnet can attract the PFCO matrix composite, indicating that the PFCO matrix composite is a ferromagnetic material. This material contains iron and nickel elements that differs from those found in existing magnetic materials such as iron, nickel and cobalt, but also differs from the alloy of iron, nickel, cobalt. It belongs to a new material has not been clearly identified, which is considered to be synthesized a new ferromagnetic material.

Insulation Performance Test

The reference material used is alumina ceramic, which has the same volume as lead matrix composite material and is cuboid. The resistance of the alumina and lead matrix composites was measured by a multimeter. The resistance of lead matrix composite material is: 30~65×2000MΩ, while the resistance of alumina is: 75~105×2000MΩ.

The result shows that the PFCO matrix composite exhibits high electric resistance and is a good insulating material.

Sem Micrograph of The Pfco Matrix Composite Fracture

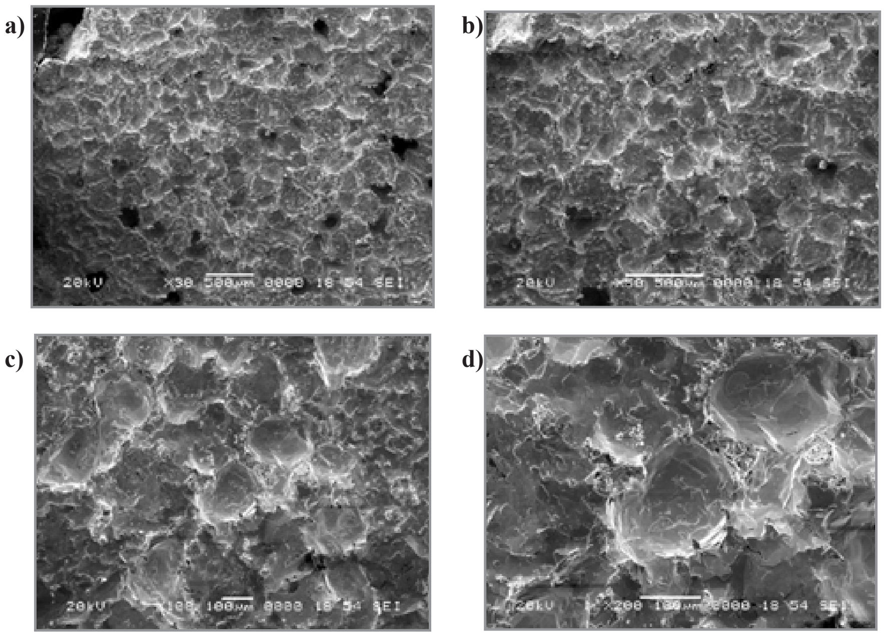
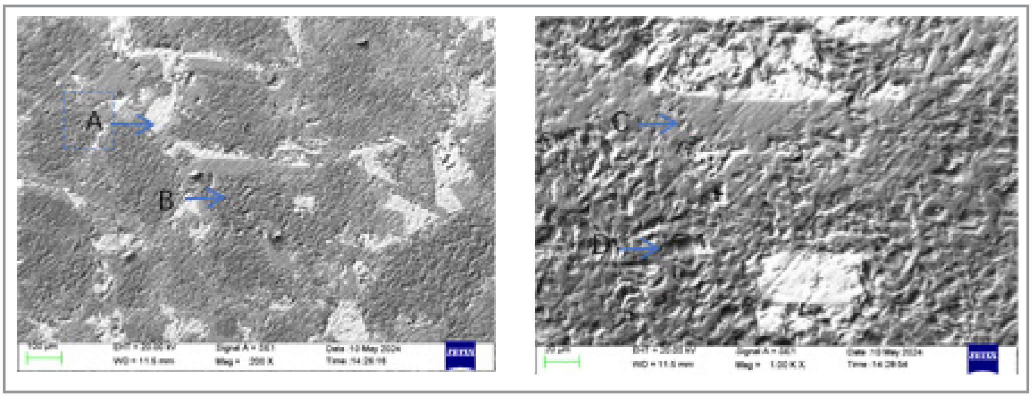


Figure. 2 SEM Micrographs of the PFCO Matrix Composite Fracture

Fig. 2 shows the SEM image of the fracture of a small piece of the PFCO matrix composite. In Fig. 2, the magnification of a), b), c), d) is 30 times, 50 times, 100 times and 200 times respectively. As illustrated in Figures 2a) and 2b), there are holes (black part) in the entire fracture tissue; As illustrated in Figures 2c) and 2d), the fracture is a dissociated floral pattern, and the local tissue is relatively evenly distributed.

There are holes in the fracture tissue in Fig. 2, indicating that the tissue is not very dense. The reason for the formation of holes may be that the maximum temperature is not enough, resulting in insufficient fluidity of the PFCO matrix composite tissue after liquefaction, and the gas in the hole is not discharged, then the hole remains in the PFCO matrix composite tissue. The fracture of the PFCO matrix composite shows a dissociation pattern, indicating that the crushing process of the PFCO matrix composite belongs to brittle fracture, and this kind of tissue is very brittle and belongs to brittle tissue.

SEM Micrographs of the Microstructure of The PFCO Matrix Composite



a) b)

Figure. 3 SEM Micrographs of the Microstructure of the PFCO Matrix Composite

As illustrated in Fig. 3, the microstructure consists of two kinds of microstructures, namely, the irregular white microstructure A and the dark microstructure B. The dark microstructure B is divided into the long and dense part C and the loose part D.

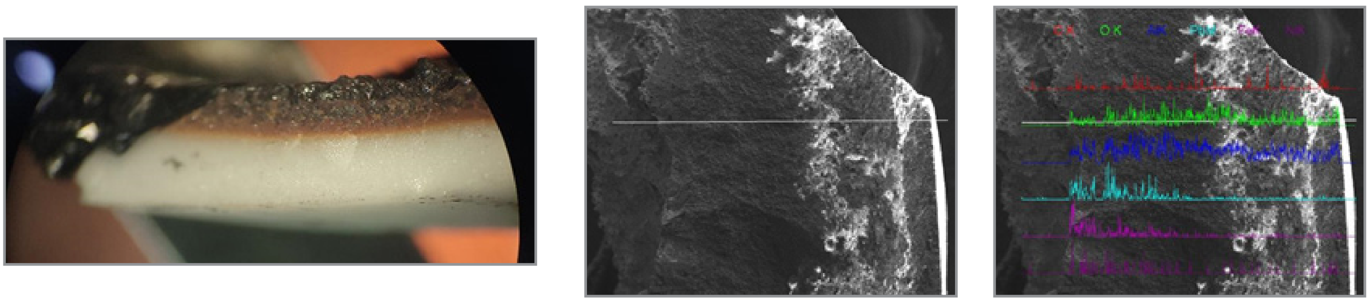
The results of energy spectrum analysis of these two microstructures are shown in Table 1. The main components of the microstructure A are Pb, O, and C, while the main components of the microstructure B are Fe, Pb, O, and C.

The results of XRD analysis above show that the product contains Fe_3O_4 and PbO, as well as an X phase. The X phase occupies the largest volume. The volume of B in the microstructure of the product is also the largest. Combining the results of XRD analysis with microstructure B, it can be considered that the X phase in XRD is the corresponding phase of microstructure B. Microstructure B contains Fe, Pb, O and C elements, then the X phase is a new phase containing Fe, Pb, O and C, which can be named the $(Pb_x, Fe_y, C_z) O_e$ (Abbreviated as PFCO) matrix.

Table. 1 Results of EDS (wt.%)

	Pb	Fe	O	C	Ni
A	68.27	10.11	11.24	9.53	0.14
B	28.52	38.12	26.19	6.18	1.12

Results of Linear Scanning of Interface



a) b) c)

Figure. 4 SEM Micrograph and Results of Linear Scanning of Interface

A piece of ceramic adhering to the PFCO matrix composite (Fig. 4a) is selected and its photograph is taken in an optical photograph (magnified 10x), which clearly shows the interface between the white ceramic and the dark PFCO matrix composite.

SEM was used to observe the fracture of this piece of ceramic adhering to the PFCO matrix composite, as shown in Fig 4b). The left side is the PFCO matrix composite and the right side is the ceramic in Fig. 4b). It was found that there is no obvious transition interface between the ceramic and the PFCO matrix composite. Linear scanning was used to analyze the composition distribution on the left and right sides of the interface, as shown in Fig. 4c). It can be seen that the compositions of Pb, Fe and Ni have a higher content on the left side of the interface, which gradually decreases from left to right, and tends to zero at the joint. The distribution of oxygen and aluminum on both sides of the interface is different from that of Pb, Fe and Ni, and the compositions of Al and O on both sides of the joint have little change.

Because the compositions of the PFCO matrix composite and the ceramic are very different, the distribution of Pb, Fe and Ni elements on both sides of the interface is very different. The source of Al is alumina ceramics, and their uniform distribution on both sides of the interface indicates that alumina ceramics are not decomposed at high temperatures, so the Al come from alumina ceramics, and in the linear scan pictures of Pb, Fe and Ni, their distribution decreases, indicating that these elements penetrate into alumina ceramics at high temperatures.

Conclusions

A kind of a (Pbx, Fey, Cz) Oe (Abbreviated as PFCO) matrix composite bonded to alumina ceramics was synthesized by mixing the powders of pure lead (Pb), pure iron (Fe), graphite (C) and pure nickel (Ni) in an atmospheric environment at a high temperature. The PFCO matrix composite is mainly composed of the (Pbx, Fey, Cz) Oe matrix, PbO and Fe3O4. The PFCO matrix composite is a ferromagnetic material and insulating material with relatively brittle properties and high hardness. The mixture of pure Lead, pure iron, graphite and pure nickel can be used as a new kind of brazing materials for alumina ceramics.

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