Effects of heat treatment on mechanical properties and microstructure of tungsten fiber reinforced grey cast iron matrix composites

*Niu Libin, Xu Yunhua, Peng jianHong, Wu Hong
(Institute of Wear Resistant Materials, College of Mechanical and Electrical Engineering, Xi’an University of Architecture & Technology, Xi’an, Shaanxi 710055, China)

Abstract: In this study, grey cast iron matrix composites reinforced by different volume fractions of tungsten fibers (Vr = 0.95%, 1.90%, 2.85%, 3.80%) were investigated in as-cast and under the heat treatment temperatures of 1,000°C and 1,100°C. The microstructure and mechanical properties of the composites were analyzed and tested by means of SEM, micro-hardness tester and three-point bend testing. The results show that with increasing of the volume fraction of tungsten fibers, the composites reinforced by the tungsten fiber have higher flexural strength and modulus than that of cast iron without reinforcement, and the flexural strength increases with the increasing of heat treatment temperatures. Due to diffusion reaction between matrix and reinforcing phases, the process of heat treatment, the number of graphite flakes in the matrix seemingly becomes lower; and some hard carbide particles are formed around the residual tungsten fibers. Not only does the hardness of both matrix and reinforcement change tremendously, but also the region of reinforcement is also extended from the original 0.11 mm to 0.19 mm in radius.

Key words: iron matrix composites; tungsten fiber; heat treatment; three-point bending; flexural strengths

Grey cast iron is the most widely used metallic material, because of its good castability, excellent machinability, relatively low cost and unique properties [1-2]. Due to the recent rapid increase of demand for high performance of equipment and machines, a great deal of research focused on how to improve the mechanical properties of the grey cast irons [3-4]. One of the significant methods used is to produce a metal matrix composite material by reinforcing it with a higher-strength material [5].

Depending on the geometry of the reinforcement, metal matrix composites are classified as particulate, short fiber or continuous fiber reinforced materials. Although particulate reinforced metal matrix composites (MMCs) have gained increasing importance, due to their high specific modulus, strength, thermal stability, and excellent wear resistance [6], fiber reinforcement to fabricate the grey cast iron matrix composite also plays an important role in the areas of aerospace, automobiles and other engineering industries. There are various processes developed for the fabrication of fibrous MMCs [7-9].

Up to today, only a few fibrous MMCs were related with the grey cast iron matrix. For example, Akdemir et al. [10] worked on the strengthening and toughening of cast iron by reinforcing it with steel fiber through heat treatment. In that process, the number of brittle graphite flakes in the matrix was decreased due to the diffusion of carbon atoms from the matrix to the steel fibers, and as a result, the mechanical properties of grey cast iron were improved. Simsir [11-12] studied on the fracture behavior of laminated metal matrix composite and tough steel fiber-reinforced grey cast iron composite. Although it is available to improve the mechanical properties through strengthening grey cast iron by reinforcement, the microstructure of grey cast iron still contains a larger number of brittle graphite flakes, which basically control the mechanical properties and confer low strength and toughness. Therefore, the principal approach of improving the mechanical properties is to decrease the carbon equivalent, which reduces the percentage of graphite flakes in the microstructure.

It is well known that tungsten carbide can be traditionally produced through the mixtures of tungsten and graphite powders under certain conditions [13]. This gives us a clue, if the graphite flakes phase in the grey cast iron matrix can react with tungsten added into matrix, and carbide can be formed. There are two distinct functions: on the one hand, the hardness and wear resistance of grey cast iron can be enhanced for the formation of tungsten carbides [14], and on the other hand, the number of brittle graphite flakes can be decreased due to the carbide formation. These results will lead to modification of
the mechanical properties of grey cast iron.

Hence, in the present research, the first objective was to produce iron matrix composite consisting of high-strength tungsten fibers and relatively low flexural-strength grey cast iron, in which the reinforcement is added into the grey cast iron matrix in the form of tungsten mesh to ensure an uniform distribution. The second objective of this study was to decrease the brittle graphite flakes by diffusion reaction between tungsten fiber and graphite flakes in the process of heat treatment. The third objective was to investigate the effect of heat treatment on the mechanical properties of the composites, especially the bend/flexural strengths.

1 Experimental procedure

1.1 Specimen fabrication

The composites consist of high-flexural-strength tungsten fiber (99.2 % purity, 0.22 mm in diameter) as reinforcement and grey cast iron (given in Table 1) as matrix. The tungsten fiber was acquired from Baoji Refractory Metal Developer Co., Ltd., Baoji, Shaanxi, China. The grey cast iron was melted using pig iron, ferrosilicon, ferromanganese, steel strip as charge materials in an acid-lined crucible induction furnace (10 Kg). By design, the strengthening of the grey cast iron is obtained by reinforcing it with tungsten fibers.

Metal-mould casting technique was used for the production of both the grey cast iron without and with reinforcement. For preparation of reinforcement, tungsten mesh was weaved with tungsten fibers as latitude and copper wires as longitude, as shown in Fig.1 (a). Then, the tungsten mesh was cut into several small patches of 100 mm × 30 mm; subsequently, small patches were cleaned of all dirt, grease for 20 min in a solution containing 4 % HNO₃, washed with alcohol and dried. In order to avoid possible movement of tungsten fibers during the casting process, the fastness of the tungsten mesh was crucial. A series of sockets were cut in the profile of metal mould and their widths were closely equal to the thickness of the tungsten fiber mesh, as shown in Fig. 1(b). Before casting, the tungsten meshes were inserted into sockets, then the mould cavity and tungsten meshes were heated up to approximately 200°C with an electric furnace. Molten cast iron was later poured into the metal mould at the temperature of 1,300–1,350°C, and silica sand was used to cover the metal mould. The specimen was taken out of the mould after it cooled to room temperature. The grey cast iron matrix composites produced using reinforcing tungsten fibers were machined to prepare the specimens with dimension of 6 mm ×10 mm ×10 mm for metallographic analysis, as shown in Fig. 2 (a). After calculation, it was determined that four composites were produced, containing different volume fractions of reinforcement, at $V_r = 0.95 \%, 1.90 \%, 2.85 \%$ and $3.80 \%$, respectively. To compare the behaviors of the base grey cast iron and the composites, the grey cast iron and the composites were cast under the same casting conditions to yield specimens with the same dimensions.

<table>
<thead>
<tr>
<th>C</th>
<th>S</th>
<th>P</th>
<th>Si</th>
<th>Mn</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.61</td>
<td>0.024</td>
<td>&lt;0.022</td>
<td>1.30</td>
<td>0.41</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>$P_r$</th>
<th>0.95 %</th>
<th>1.90 %</th>
<th>2.85 %</th>
<th>3.80 %</th>
</tr>
</thead>
</table>

Fig. 1: Schematic diagrams showing original tungsten mesh (a) and metal mould and inserting location of tungsten mesh (b)

Fig.2: Schematic illustration of specimens for metallographic analysis (a) and three-point bending (b)
Three-point bend specimens were produced from the grey cast iron and the composites to the dimension of 6 mm × 10 mm × 40 mm. In the bend test, the ratio of span length to width used in the determination of the flexural strength was kept to be equal to five (5), as shown in Fig. 2(b).

To evaluate the effect of heat treatment on the mechanical properties and microstructures, the as-cast specimens with and without reinforcement were heated in an electric muffle furnace to 1,000°C and 1,100°C, respectively, at a rate of 20°C/min. The specimens were held at the specified temperatures for 4 hours followed by furnace cooling to room temperature.

1.2 Testing

1.2.1. Flexural strength and Flexural modulus

Three-point bend test was applied to determine the flexural strengths of the grey cast iron and the composite materials. The tests were performed using a universal test machine (Computer servo control materials testing machines HT-2402) under stroke-control with a crosshead speed of 0.01 mm/s. The span length was five times greater than that of the specimen width, see Fig. 2(b). The maximum bending load ($P_{\text{max}}$), maximum deflection ($\delta_{\text{max}}$), flexural strength ($\sigma_b$), and flexural modulus ($E_b$) were determined using the equations given below. The test was repeated three times for each specimen, and the average of the test results was used in the evaluation.

$$\sigma_b = \frac{3PL}{2bh^2} \quad (1)$$

$$E_b = \frac{PL}{4bh}\delta \quad (2)$$

Where, $P$ is the maximum load, $b$ is the thickness of the specimen (10 mm), $h$ is the width of the material (6 mm), $L$ is the span length (30 mm), and $\delta$ is the deflection at maximum load.

1.2.2 Metallographic examination

Metallographic specimens were prepared using conventional procedure and etched with 2% nital reagent. Scanning electron microscope (Model: VEGA/TESCAN) equipped with energy-dispersive spectrum and X-ray diffraction (XRD) (Model: D8 DIS-COVER) were used to study the microstructures and phase constitutes.

1.2.3 Hardness

The hardness values for both cast iron and reinforced composites were measured on the as-cast and the heat treated specimens. Brinell hardness tests (Model HBRV-187.5) at the 150 kg load were performed on the side of matrix for all specimens. Micro-hardness ($HV_{0.1}$) was also measured using a Tukon 2100B tester with dwelling time of 10 s on the reinforcement side for the composites.

2 Results and discussion

2.1 Flexural strength and modulus

Load versus deflection curves of the composite materials with different volume fractions ($V_r = 0.95\%$, $1.90\%$, $2.85\%$, and $3.80\%$) and the grey cast iron were obtained under the as-cast condition (Fig. 3). The $P_{\text{max}}$ and $\delta_{\text{max}}$ values increase with increasing of volume fraction of the tungsten fibers. Grey cast iron has strain of 0.47mm at $P_{\text{max}}$. The maximum strain was achieved at $P_{\text{max}}$ for the $V_r = 3.80\%$ composite. The strain ($P_{\text{max}}$) of the as-cast composite with $V_r = 3.80\%$ is 8.75% higher than that of grey cast iron. Similar increments in the strain ($P_{\text{max}}$) were observed for the heat treated composites at 1,000°C and 1,100°C, and similar curves were obtained for the specimens heat treated at 1,000°C and 1,100°C. It can be concluded that the ductility of the grey cast iron can be improved by reinforcement with tungsten fibers.

According to equation (1), the flexural strengths of the grey iron and composite materials with four different volume fractions ($V_r = 0.95\%$, $1.90\%$, $2.85\%$, and $3.80\%$) were determined both under the as-cast and heat treatment conditions at 1,000°C and 1,100°C. The variations in the flexural strengths versus volume fraction curves obtained for both the as-cast and the heat treatment conditions are shown in Fig. 4.
strength values was caused by the differences in casting conditions and chemical composition etc. The flexural strength values of the composites with \( V_r = 0.95 \% \), 1.90 \%, 2.85 \%, and 3.80 \% were calculated, and found to be 3.00 \%, 7.43 \%, 9.91 \%, and 11.6 \% greater than that of the grey cast iron, respectively. The maximum flexural strength was 285 MPa which was obtained for the composite with \( V_r = 3.80 \% \). The present results show that the flexural strength of the grey cast iron can be increased by reinforcing with high-strength tungsten fiber and by increasing the volume fraction of the reinforcement.

As observed in Fig. 4, the flexural strength values for specimens without reinforcement after heat treated at 1,000°C and 1,100°C had no distinct change, as compared with that for specimen in the as-cast condition. Under the heat treatment condition at 1,000°C, the grey iron without reinforcement had a flexural strength of 259 MPa, while the flexural strengths of composites with \( V_r = 0.95 \% \), 1.90 \%, 2.85 \%, and 3.80 \% were 4.97 \%, 9.39 \%, 11.90 \% and 12.85 \% greater than that of the grey cast iron, respectively. For the heat treatment condition at 1,100°C, the grey cast iron without reinforcing had a flexural strength of 263 MPa, and the flexural strengths of the four composites (with \( V_r = 0.95 \% \), 1.90 \%, 2.85 \%, and 3.80 \%) were respectively 6.35 \%, 10.7 \%, 13.3 \% and 16.7 \% higher than that of grey cast iron. The composite with \( V_r = 3.80 \% \) heat treated at 1,100°C achieved the maximum flexural strength up to 307 MPa.

Figure 5 shows the variation of the flexural modulus with the volume fraction of reinforcement under the as-cast condition. The flexural modulus of the grey cast iron was increased non-linearly by the reinforcement with tungsten fibers. As compared to the flexural modulus of 26 GPa for the gray iron without reinforcement, the flexural modulus of composites with \( V_r = 0.95 \% \), 1.90 \%, 2.85 \%, and 3.80 \% were respectively 8.73 \%, 21.7 \%, 25.0 \% and 37.9 \% higher. It is noted that the maximum increment in flexural modulus was obtained in the composite with \( V_r = 3.80 \% \).

2.2 Microstructures

Figure 6 (a) through (f) present the microstructures of the

![Fig. 5: Variation of flexural modulus versus volume fraction of reinforcement under the as-cast condition](image)

![Fig. 6: Microstructures of reinforcement in composite, (a, b) as-cast; (c, d) at 1,000°C; (e, f) at 1,100°C.](image)
composites under both the as-cast and the heat treatment conditions. Under the as-cast condition, the graphite flakes in the matrix of the composite were thick and randomly oriented with extremely sharp tips, and their lengths varied from 0.01 mm to 0.1 mm. The thickness of transition region between reinforcement and matrix was 5 µm. According to the results of EDS, some net-like structures, cementites, were identified, which were formed during the casting process.

It can be seen from Fig. 6(c) that, after heat treated at 1,000 °C, the cementites in the grey cast iron side were dissolved, and the original morphology of reinforcement was extremely changed into many particles/fragments (still called as reinforcement). Although, as compared with the as-cast condition, the maximum lengths of the graphite flake did not change considerably with increasing of heat treatment temperature, the number of the shorter graphite flakes in the matrix became decreasing, resulting in removal of some brittle graphite flakes. In addition, the graphite flakes becoming thinner and oriented also cause a rise in the flexural strength \[11\]. In this case, due to the high temperature and consequently higher diffusion rate, a certain chemical reaction occurred between matrix and reinforcement, and some carbide particles were formed surrounding the tungsten fiber, as shown in Fig. 6(d). Based on the XRD analysis result, the particles can be detected primarily to be WC (see Fig.7).

After heat treated at 1,100 °C, tungsten fiber completely transferred into particles, and the center of tungsten fiber was substituted by metal matrix for over-diffusion reaction (Fig. 6e). On the other hand, the number of particles was much higher than that heat treated at 1,000 °C (Fig. 6f), and the distribution area of particles was larger than the area of original tungsten fiber section. Similarly, according to the result of XRD, the particles were primarily WC and M₆C (see Fig.7). From the above analysis, it is concluded that during heat treatment, the tungsten fiber in the composites can react with the matrix, especially with the graphite carbon, and the WC and M₆C carbides form due to the diffusion of elements along the crystal boundary of grey cast iron. The number and distribution region of particles are increased with the increasing of the heat treatment temperature, leading to improvement of the mechanical behaviors such as hardness, wear resistance and so on. On the other hand, the increase in flexural strength is attributed to the decrease of the number of graphite flakes raised by the diffusion reaction in the process of the heat treatment.

2.3 Hardness

Figure 8 shows the Brinell hardness variations for the grey iron and for cast-iron side of composite with the volume fraction of the reinforcement. Under the as-cast condition, the hardness value of the grey iron without reinforcement was measured to be 247 HB, and the hardness value of the cast-iron side of composite increased slightly with the increasing of the volume fraction of the reinforcement. However, under the heat treatment conditions, there was a distinct increase in the hardness values as the volume fraction of reinforcement increased. For the specimen of the grey iron with a reinforcement of \(V_r = 3.80\%\), the hardness of cast-iron side at 1,000 °C and 1,100 °C was 11.1 % and 14.4 % higher than those of the specimens under the as-cast condition, respectively.

It may be concluded that the hardness values of cast-iron side increase with the increasing of the volume fraction of reinforcement, and that the heat treatment results in a rise in the hardness of the grey cast iron and cast-iron side of the composite.
composite. All these appear to be attributed to chemical reaction between carbon and tungsten and the formation of the carbide particles with higher hardness such as WC and M₆C, which is consistent with analysis of microstructure.

For future work, it is expected that more suitable volume fraction of the tungsten fiber can be chosen so that most of graphite flakes are removed by the reaction between graphite and tungsten, and much more carbides will be produced during the heat treatment. Furthermore, by selecting the appropriate parameters such as casting condition, tungsten fiber diameter and heat treatment condition, iron matrix composites with high strength and flexural modulus can be produced cost-effectively.

3 Conclusions

(1) In as-cast condition, the flexural strength of the grey cast iron composite reinforced by tungsten fiber increases with the increase of volume fraction of reinforcement. When the volume fraction \( V_r \) is at 3.80 %, the maximum flexural strength is up to 285 MPa.

(2) For the same volume fraction of reinforcement, the composites after heat treatment have the flexural strength higher than the composites under the as-cast condition, and the flexural strength increases with the increasing of heat treatment temperature.

(3) Under the as-cast condition, the flexural modulus of the composites with \( V_r = 0.95 \% \), 1.90 \%, 2.85 \%, and 3.80 \% was respectively 8.73 \%, 21.7\%, 25.0 \% and 37.9\% higher than that of the base grey cast iron.

(4) During heat treatment, the tungsten fibers in the composites react with the matrix, especially with the graphite carbon, forming WC and M₆C carbides due to the diffusion of elements. The number of carbide particles increases with the increasing of heat treatment temperature, which make the distribution region enlarged from 0.11 mm to 0.19 mm in radius. Moreover, the change in the hardness of both matrix and reinforcement takes place because of the diffusion reaction between matrix and reinforcement.

References


The work was fully supported by the Key Innovation Plan in Science & Technology, Shaanxi Province, China (Project No. 2004ZKC05-02) and the Research Center for "13115" Innovation Engineering in Science & Technology, Shaanxi Province, China (Project No. 2007ZDGC-17).