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Warm compacted NbC particulate reinforced iron-base composite(Ⅰ)①

——Effect of fabrication parameters

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[Abstract] NbC was used as reinforced particles in the fabrication of iron-base composite. Ball milling was introduced to overcome the problems of agglomeration and powder separation during powder mixing. After ball milling, the fine NbC particles are embedded on the surface of iron particles and evenly distributed in the mixed powders. Warm compaction was used not only to increase the green density but also to improve the formability of the mixed powder and to improve the compact's green strength to facilitate handling. The influences of fabrication parameters such as ball milling time, annealing temperature and time, warm compaction temperature, sintering temperature and sintering time were studied. Compacts with a relative sintered density of 97% and a tensile strength of more than 800 MPa can be obtained by using a ball milling time of 5 h, an annealing temperature of 800 ℃, a compaction pressure of 600 MPa, warm compaction temperature of 120 ℃, sintering temperature of 1 280 ℃, and sintering time of 80 min. The shrinkage at this sintering condition was approximately 4.3%.

[Key words] iron-base composite; NbC particulate; warm compaction powder metallurgy

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1 INTRODUCTION

Metal matrix composites (MMCs) combines the advantages of different materials. It is a hot spot in the past decades. Powder metallurgy (PM) is a convenient and effective way to produce MMCs, but research on PM iron base MMCs is limited. Although ceramic particulates, such as carbides, nitrides and oxides reinforced iron or steel-base composite materials have attracted some attention[1~3], very seldom PM iron-base composite with good mechanical properties and tribological behaviors were reported. The introduction of ceramic particulates into metallic powders will unavoidably lowers the compressibility and formability of the mixed powder due to the physical and mechanical properties of the hard particulates. In order to solve these problems, in this study, warm compaction was introduced in preparing the iron-base composite. Warm compaction is a simple and effective way to produce high density PM materials with high green strength[4~7]. It has attracted great attention since 1996 in our country[8~12]. Another problem in PM MMCs fabrication is the agglomeration and powder separation following the mixing of different powders. Ball milling of the mixed powders was introduced to minimize these problems. The aim of this study is to develop an iron-base composite with high density, good mechanical properties and tribological behaviors for high performance applications.

2 EXPERIMENTAL

High purity water atomized iron powder and NbC particulates with an average particle size of 75 μm and 5 μm, respectively, were used as starting materials. Fine powders of graphite, carbonyl Ni, electrolytic Cu, Mo and phosphorus iron with 25% P (mass fraction) were used as alloying elements. Composition of the mixed powder in mass fraction was 0.5%~2.0% Cu, 2%~4% Ni, 0.5%~1.0% Mo, 0.6%~1.0% C, 0.4%~0.8% P and 0 to 15% NbC. Unless mentioned, all compositions were reported in mass fraction through out this paper. Additional 0.25% of polymeric lubricant was mixed with the powder in a stir type ball milling machine using steel balls of 5 mm in diameter. The ball to powder ratio was 10∶1 and the milling time was ranging from 0 to 7 h. The ball-milled powder was then mixed in a rotary mixing machine for 0.5 h. Annealing temperatures of 600, 700 and 800 ℃ were used to anneal the mixed powder for 5 h to increase the powder compressibility. The annealed powder was warm compacted into standard tensile test bars (GB 7963-1987) at a temperature ranging from ambient to 140 ℃ with pressure of 500, 600 and 700 MPa in a heated steel

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mold. Emulsified polytetrafluoroethylene (PTFE) was brushed on the inner die wall for lubrication. These samples were degassed at 400 ℃ and pre-sintered at 800 ℃ in the pre-heating chamber of a pusher type furnace, with all its chambers protected by a hydrogen-nitrogen reducing atmosphere. Sintering was carried out at temperatures ranging from 1 150 ℃ to 1 300 ℃ for a sintering time ranging from 40 to 100 min in the sintering chamber, then held at 650 ℃ in the cooling chamber and subsequently cooled to room temperature. Green density, sintered density and tensile strength were measured. Microstructures were analyzed by optical microscopy and scanning electron microscopy (SEM). Chemical compositions were analyzed using energy dispersive X-ray spectroscopy (EDX) equipped in SEM.

Relative density was used instead of density and it is defined as: relative density = (compact density/pore free density) × 100%.

Sample shrinkage was measured by micrometer according to GB/T5159—1985. Shrinkage percentage (Δd_{GS}) is defined as: $\Delta d_{GS} = [(d_S - d_G)/d_G] \times 100\%$, where d_G and d_S are the sizes of the green compact and sintered compact, respectively.

3 RESULTS

3.1 Powder treatment

Fig. 1 shows the average particle sizes versus ball milling time. At the beginning of the ball milling, the particle size increases as the milling time increases, then, after 1 h of milling the particle sizes drop with increasing milling time and become stable after 7 h of milling. Fig. 2 is a SEM micrograph showing the NbC particles embedded on the surface of the iron powder particles. Composition analysis using EDX confirms this result. Fig. 3 shows the effects of ball milling time (without annealing) and annealing temperature (after 5 h of ball milling) on the relative green density of the Fe-0.6P-0.8C-10NbC compacts using a compaction pressure of 600 MPa. For powder without annealing, the relative density of the green compacts decreases as the milling time increases. The compressibility of the powder increases as the annealing temperature increases and the relative density reaches a value of 75% after annealing at 800 ℃. Considering the effectiveness and the cost factor, a milling time of 5 h and an annealing temperature of 800 ℃ were used in the rest of our experiments unless otherwise specified.

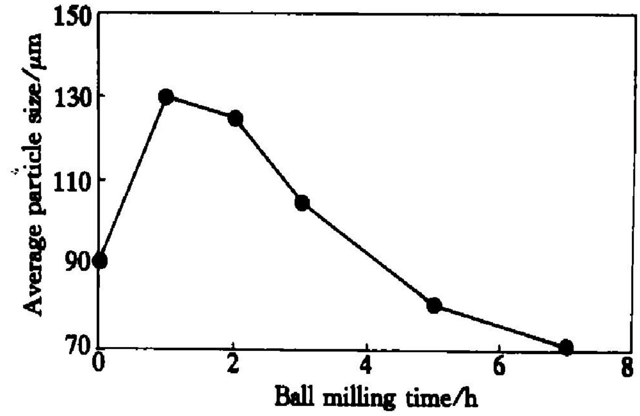

Fig. 1 Average particle size versus ball milling time

Fig. 2 NbC embedded on surface of iron powder

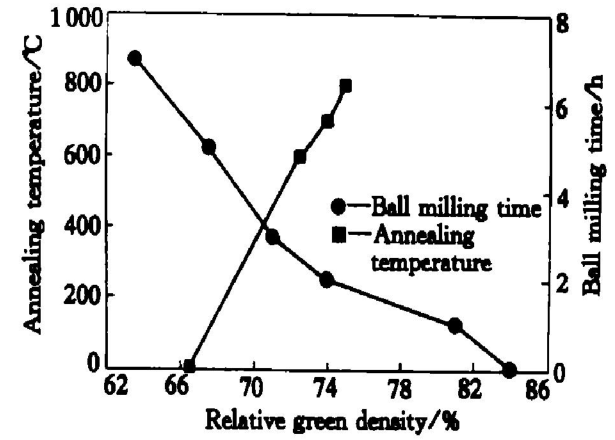

Fig. 3 Effects of ball milling time (without annealing) and annealing temperature (after 5 h of ball milling) on relative green density of compacts using a compaction pressure of 600 MPa

3.2 Warm compaction of NbC reinforced iron-base composite

Fig. 4 shows the effect of compaction pressure on the relative green density of compacts with different NbC contents. The relative density increases with increasing compaction pressure, but for a pressure larger than 700 MPa the increase in relative density diminishes. Fig. 5 compares the effects of warm compaction at 120 ℃ and cold compaction on the relative green density of compacts with different NbC contents using a compaction pressure of 600 MPa. It shows that the warm compacted samples have higher relative densities and the densities decrease with increasing NbC content.

Fig. 6 shows the effect of warm compaction temperature on the relative green density of the compacts

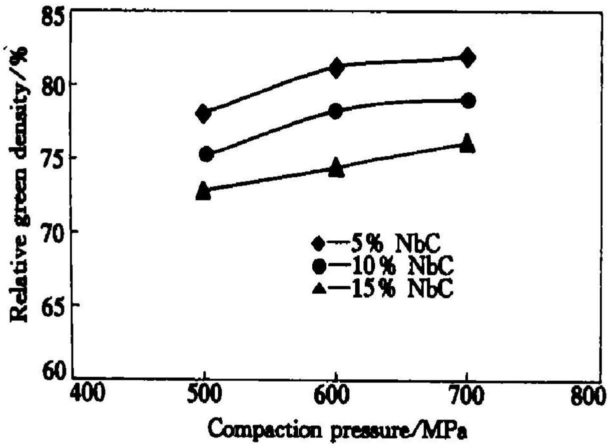

Fig. 4 Effect of warm compaction pressure at 120 ℃ on relative green density of compacts with different NbC contents

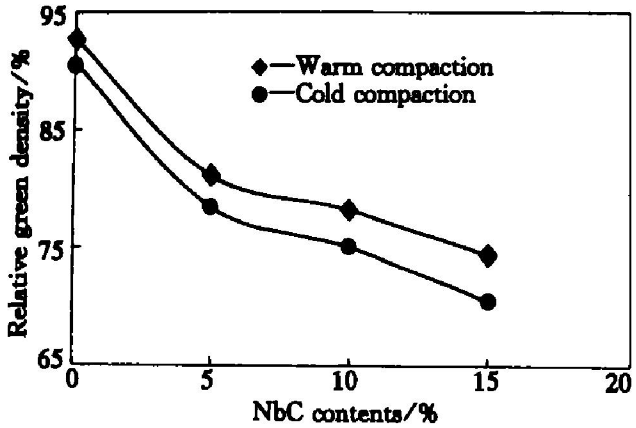

Fig. 5 Effects of warm compaction at 120 ℃ and cold compaction on relative green density of compacts with different NbC contents at compaction pressure of 600 MPa

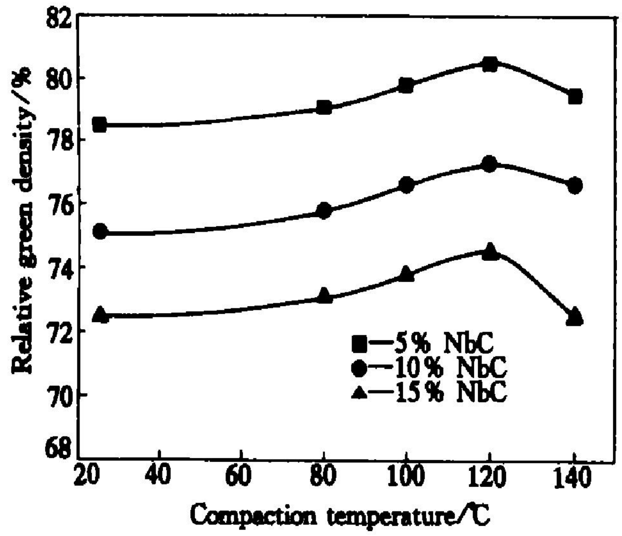

Fig. 6 Effect of warm compaction temperature on relative green density of compacts with different NbC contents at compaction pressure of 600 MPa

with different NbC contents. The relative density of the compacts increases with increasing warm compaction temperature and it reaches the maximum at 120 ℃ then decreases. The higher the NbC content the lower the relative density of the compact. Unless mentioned, the compaction pressure and temperature used in the following experiments are 600 MPa and 120 ℃, respectively.

3.3 Sintering of NbC reinforced iron-base composite

Fig. 7 shows the effect of sintering temperature on the relative density and the shrinkage of the sintered sample with 10% NbC using a sintering time of 80 min. The shrinkage increases rapidly as the sintering temperature increases to higher than 1 160 ℃ then leveles off at approximately 1 260 ℃. While, the relative density increases rapidly with the increase of sintering temperature then leveled off at approximately 1 160 ℃ also. Fig. 8 shows the effect of sintering time on the relative density and the shrinkage of the sintered sample with 10% NbC at a sintering temperature of 1 280 ℃. As shown in Fig. 8, the relative density and the shrinkage increase rapidly as the sintering time increases then they level off after 80 min of sintering.

In this study, compacts with a relative sintered

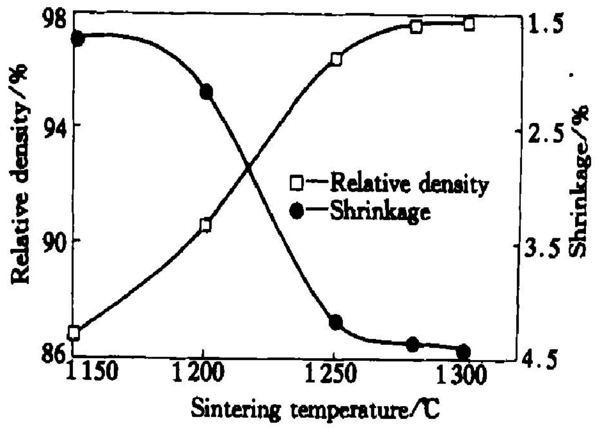

Fig. 7 Effect of sintering temperature on relative density and shrinkage of compacts with 10% NbC sintered for 80 min

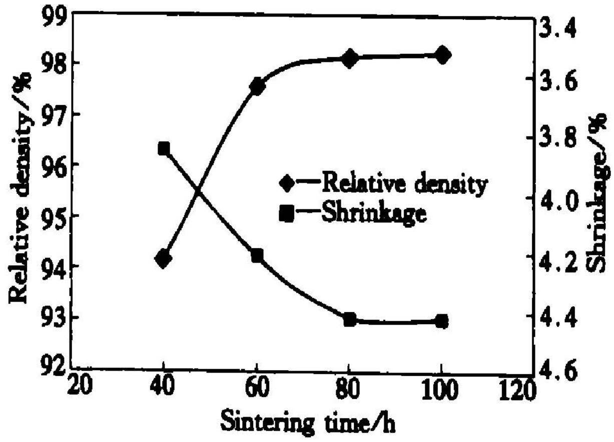

Fig. 8 Effect of sintering time on relative density and shrinkage of compacts with 10% NbC sintered at 1 280 ℃

density of 97% and a tensile strength of more than 800 MPa can be obtained by using a ball milling time of 5 h, an annealing temperature of 800 ℃, a compaction pressure of 600 MPa, warm compaction temperature of 120 ℃, sintering temperature of 1 280 ℃, and sintering time of 80 min. The shrinkage at this sintering temperature and time is approximately 4.3%.

4 DISCUSSION

Agglomeration and powder separation are commonly found in the mixing of powders due to the differences in particle sizes, shape, densities and surface characteristics. Ball milling of reinforced particles with metal powders is an effective way in solving problems which are encountered in conventional mixing[13-15]. As shown in Fig. 1 the average particle size increases with ball milling time. At the initial stage of milling metallic particles change to flaky in shape and their sizes increase due to particle deformation and cold welding. After 1 h of milling the particle sizes drop as milling time increases due to the particle plasticity decreases. The work hardening of the particles makes them brittle and subject to break during the ball milling. After 7 h of milling, the breaking and the cold welding of powder particles reach an equilibrium and the particle size becomes stable. There is no observable reaction taking place during the milling. SEM analysis and EDX results confirm that NbC particles are embedded on the surface of iron powder particles and thus helping the even distribution of NbC in the mixed powder. This evenly mixed powder is vital in obtaining high quality metal matrix composite. Due to the low compressibility of NbC, the relative green density of the compacts decreases as the NbC content increases. As the milling time increases the relative density of the green compacts decreases and no qualified compacts can be produced even a compaction pressure of 700 MPa is used. The high NbC contents and the work hardening effect in the ball milling process will dramatically reduce the compressibility of the powder, therefore annealing is required. The compressibility of the powder can be enhanced with higher annealing temperature.

As shown in Fig. 5, at 10% NbC there is a 3.2% increase in relative density if die wall lubricated warm compaction was used instead of cold compaction. The reason to employ die wall lubricated warm compaction is not only to increase the density but also to overcome the problems of crack formation during the compaction and increase the green strength to facilitate the handling of the green compacts. Our experiments show that cracks are formed on the compact surface after forming by cold compaction, however, no such phenomenon can be observed if warm compaction is employed. The optimal warm compaction temperature is found to be 120 ℃ under our experimental condition. This optimal temperature corresponds to the phase transition temperature of the lubricant used in this study.

The increase in sintering temperature will make transient liquid phase sintering possible and enhance the diffusivity of the alloying elements. The large shrinkage percentage and the increase in relative density are due mainly to the presence of liquid phase. The increase in sintering time will allow enough time for the inter-diffusion of elements and thus enhance the quality of the sintered compacts.

5 CONCLUSIONS

1) The introduction of ball milling solve the problems of agglomeration and powder separation during the powder mixing procedures. After ball milling, the fine NbC particles embed on the surface of iron powder particles and evenly distribute in the mixed powder. Although ball milling will worsen the compressibility of the powder, annealing at 600 ℃ to 800 ℃ can improve the compressibility of the ball milled powder.

2) Both green density and green strength of the iron-base NbC particulate reinforced composite are enhanced by die wall lubricated warm compaction. The optimal warm compaction temperature is found to be 120 ℃ under our experimental condition. In this study compacts with a relative green density of 75% can be obtained by using a ball milling time of 5 h, an annealing temperature of 800 ℃, warm compression temperature of 120 ℃ and a compaction pressure of 600 MPa. Compacts with a relative sintered density of 97 % and a tensile strength of more than 800 MPa can be obtained by sintering at a temperature of 1 280 ℃ for 80 min. The shrinkage at this sintering temperature and time is approximately 4.3%.

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