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Warm compacted NbC particulate reinforced iron-base composite(II) ⁽¹⁾ —Microstructure and properties

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[Abstract] Effects of different sintering temperature and sintering time on the relative density of the sintered compacts were studied to obtain the optimal sintering parameters for the fabrication of NbC particulate reinforced iron base composite. With optimal sintering temperature of 1 280 °C and sintering time of 80 min, wear-resisting, high density NbC particulate reinforced iron base composites can be obtained using warm compaction powder metallurgy. The microstructure, relative density, mechanical properties and tribological behaviors of the sintered composites were studied. The results indicate that the mechanical properties of the sintered compacts were closely related to the sintered density. The iron base composite materials with different combinations of mechanical properties and tribological behaviors and tribological behaviors were developed for different applications. One of the developed composite, which contains 10% NbC, possesses a high strength of 815 M Pa with a remarkable friction and wear behaviors. The other developed composite, which contains 15% NbC, possesses a lesser strength of 515 M Pa but with excellent friction and wear behaviors.

[Key words] iron base composite; NbC particulate; mechanical property; tribological behaviors [CLC number] TF 12 [Document code] A

1 INTRODUCTION

The biggest problems in the fabrication of particulate reinforced metal matrix composites (MMCs) are the even distributions of reinforce particles and the wetting ability between the matrix and the reinforced particles. These problems will also be encountered in powder metallurgy (PM), especially when liquid phase is involved. PM is a simple and economical way to produce net-shape or near-net-shape parts. The mixing of different powders in PM can provide us a wide range of composition selection in the MMCs. Our aim is to develop a particulate reinforced ironbase composite with good mechanical properties and tribological behaviors using PM technique. The even distribution of reinforced particles and the good binding between the particles and the matrix are essential elements to obtain composites with good mechanical properties and tribological behaviors. Different ceramic particulates reinforced MMCs have been studied^[1~7]. Carbides, oxides and nitrides such as SiC, WC, TiC, VC, Mo₂C, Al₂O₃ and TiN are commonly used in MMCs. However, most of these carbides are not stable with iron at high temperature, therefore they are not suitable for high temperature PM processing. The wetting angle between iron and Al₂O₃ are quite large and it is not suitable for PM processing that involves liquid phase. Our research indicates that SiC and Al₂O₃ are not good candidates to be used as reinforced particles in PM iron base MMCs^[8~10]. To overcome the problems mentioned above, we use NbC particulate, which has a density of 7. 85 g/cm^{3[11]} and a wetting angle of 21° with Fe P-C alloy at 1 280 °C^[12], as the reinforced particles. The similar densities of NbC and Fe minimize the powder separation during the powder mixing and the small wetting angle provides a good binding interface between the NbC and the iron matrix.

2 EXPERIMENTAL

High purity water atomized iron powder and NbC particulate with an average particle size of 75 μ m and 5 μ m, 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 powders in mass fraction were 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 20% NbC, with Fe as balance. 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.

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The ball to powder ratio was 10: 1 and the milling time was 5 h. The ball-milled powder was then mixed in a rotary mixing machine for 0.5 h. Annealing temperature of 800 °C was used to anneal the mixed powder for 5 h to increase the powder compressibility. The powder was warm compacted into standard tensile test bars (GB 7963-1987) and impact toughness test bars (GB 9096-1988) at a temperature of 120 °C and a pressure of 600 MPa in a heated steel mold. Emulsified Polytetrafluoroethylene (PTFE) was brushed on the inner die wall for lubrication. These samples were degassed at 400 °C and pre-sintered at 800 $^{\circ}$ C 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 °C to 1 300 °C for a sintering time ranging from 40 to 100 min in the sintering chamber then hold at 650 $^{\circ}$ C in the cooling chamber and subsequently cooled to room temperature. Sintered density, tensile strength(σ_b), elongation (δ), impact toughness (α_k) and hardness were measured. Microstructures, fracture surface and wear surface were analyzed by optical microscopy and scanning electron microscopy (SEM). Chemical compositions were analyzed by 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) $\times 100\%$.

Friction coefficient (μ) and wear behaviors of the sintered samples were measured by MM200 tribotester using a rotating speed of 200 r/min and a load of 980 N. The total linear sliding distance was 2.5 km. 20[#] lubricating oil was used as lubricant. The total linear sliding distance was 2.5 km. The sample size was 12 mm × 10 mm × 10 mm and the counterpart was GCr15 steel ring with a hardness of HRC 58-60.

3 RESULTS

Fig. 1 shows the tensile strength and elongation of sintered compacts with 10% NbC (HGFF1) versus sintering temperature for a sintering time of 80 min. The tensile strength and elongation increase with increasing sintering temperature and it reaches the maximum at approximately 1 290 °C. Fig. 2 shows the hardness and impact toughness of sintered HGFF1 versus sintering temperature for a sintering time of 80 min. The hardness and impact toughness increase with increasing sintering temperature and it reaches the maximum at approximately 1 290 °C also.

Fig. 3 shows the tensile strength and hardness of sintered HGFF1 versus sintering time for a sintering temperature of 1 280 ℃. The tensile strength and hardness increase with increasing sintering time and they level off after 80 min of sintering.



Fig. 3 Effect of sintering time on tensile strength and hardness of HGFF1 sintered at 1 280 ℃

Fig. 4 shows the effect of NbC contents on the relative density of warm compacted and cold compacted samples sintered at 1 280 $^{\circ}$ C for 80 min. The relative densities increase with increasing NbC contents and level off at approximately 10% NbC for both

warm compacted and cold compacted samples. Fig. 4 shows that the warm compacted samples always show a higher relative density. Fig. 5 shows the tensile strength and hardness of compacts sintered at a temperature of 1 280 $^{\circ}$ C for a sintering time of 80 min versus NbC contents. The tensile strength and hardness increase with increasing NbC contents and the tensile strength reaches the maximum at approximately 10% NbC then decrease.



Fig. 4 Effect of NbC contents on relative density of warm compacted and cold compacted samples sintered at 1 280 °C for 80 min



Fig. 5 Effect of NbC contents on tensile strength and hardness of compacts sintered at 1 280 °C for 80 min

Figs. 6 and 7 are SEM micrographs showing the distribution of NbC particles in the iron matrix and the excellent binding between the particles and the matrix in HGFF1, respectively. Voids can be seen in the vicinity of the NbC particles but the overall binding between the particle and the matrix is fairly good.

Fig. 8 is SEM fractography showing the brittle fracture of HGFF1 sintered at 1 280 °C for 80 min. Fig. 9 shows the wear surface morphology of samples containing 15% NbC (HGFF2) sintered at 1 280 °C for 80 min. Table 1 shows some physical and mechanical properties, and tribological behaviors of the sintered samples. Although the densities of HGFF1 and HGFF 2 are very close to each other, HGFF 1 has



Fig. 6 SEM micrographs showing distribution of NbC particles in iron matrix of HGFF1



Fig. 7 SEM micrographs showing excellent binding between particles and matrix in HGFF1



Fig. 8 SEM fractography showing brittle fracture of HGFF1 sintered at 1 280 ℃ for 80 min

better mechanical properties and HGFF2 has better tribological behaviors.

4 DISCUSSION

As reported in Part I of this paper^[13], the relative density of the sintered compacts increase with increasing sintering temperature and time then level



Fig. 9 Wear surface morphology of HGFF2

off. As shown in Fig. 1 to 3 the mechanical properties of the sintered compacts increase with increasing sintering temperature and time then level off also. These results demonstrate the close relationship between the mechanical properties and the sintered density, especially for the strength. High sintering temperature and long sintering time are not preferred not only because of the higher cost but also it may cause shape deformation and grain coarsening. Therefore, the optimal sintering temperature and time were chosen as 1 280 °C and 80 min, respectively. Fig. 5 shows that tensile strength and hardness of the sintered compacts increase with increasing NbC contents. The tensile strength reaches the maximum at approximately 10% NbC then decreases. To take advantages of the maximum tensile strength, samples containing 10% and 15 % NbC were prepared in order to study their friction and wear behaviors. Our results show that NbC are evenly distributed in the Fe matrix and very small amount of voids can be found at the particle matrix interface, especially for the large and irregular NbC particles. Fortunately, these voids are controlled to a reasonably low level. The good binding interface between NbC and the matrix provides the good mechanical properties for the tested composites.

In this study, the iron-base composite materials with different tribological behaviors were developed for different applications. HGFF1 and HGFF2 are the two high density, high strength composites with 10% and 15% of NbC particulates, respectively, reinforced in the Fe-Cu-Ni-Mo alloy matrix. Although their sintered densities are approximately the same, the HGFF1 has better mechanical properties while HGFF2 has better tribological behaviors. As shown in Fig. 8 and Table 1, HGFF1 and HGFF2 are materials with low toughness. Although HGFF2's toughness is lower than that of HGFF1, it has better tribological behaviors due to its higher NbC contents. After a total linear sliding distance of 2.5 km in the wear test, only 0.032 mm³ of the sintered materials was lost and there is no serious wear can be observed, as shown in Fig. 9 and Table 1. Minor plough and adhesion are the dominated characteristics in the wear surface.

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5 CONCLUSIONS

The similar densities of the NbC and iron minimize the powder separation during the powder mixing. The small wetting angle of Fe on NbC provides a good binding interface between the NbC and the iron matrix after the sintering. The optimal NbC contents in the composite were found in the range of $10\% \sim$ 15%. Mechanical properties of the PM iron-base composite are closely related to the sintering temperature and time. With optimal sintering temperature of 1 280 °C, sintering time of 80 min and other fabrication parameters, the high wear-resisting performance, high density NbC particulate reinforced ironbase composites were obtained in this study using warm compaction powder metallurgy. In this study, iron-base composite materials with different tribological behaviors were developed for different applications. HGFF1, which contains 10% NbC, possesses a high strength of 815 MPa and remarkable friction and wear behaviors, is suitable for general purpose wearresisting parts. HGFF2, which contains 15% NbC, possesses a lower strength of 515 MPa but excellent friction and wear behaviors, is suitable for wear-resisting parts used in extreme cases.

 Table 1
 Some physical and mechanical properties, and tribological behaviors of sintered samples

Sample	Relative green density/%	Relative sintered density/ %	σ _b / M Pa	δ/ %	$\begin{array}{c} \alpha_k / \\ (\ J^{\bullet} \ \mathrm{cm}^{-2}) \end{array}$	Hardness (HRC)	Wear volume/ mm ³	Wear rate/ (10^{-8} mm ³ • N ⁻¹ •m ⁻¹)	μ
HGFF1	78.3	97.7	815	1.5	11	33	0.065	2. 596	0. 093 6
HGFF2	74.8	98.2	515	-	6	60	0.032	1. 278	0. 084 5

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