Infiltration sintering properties of Ni-4B-4Si(wt.%) alloy powders

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Abstract. The Ni-4B-4Si(wt.%) alloy powders were infiltrated into the nickel skeletons, the effects of sintering temperatures (1050-1150 °C) and skeletons (loose and compact nickel powders) on the microstructures and hardness of the sintered alloys were investigated. The Ni-B-Si alloy sintered at 1100 °C consisted of γ-Ni and Ni₃B, and Si mainly solid soluted in the γ-Ni. The loose nickel powders favored to the infiltration of Ni-B-Si liquid alloy into the nickel skeletons, the sintered alloys exhibited dense microstructures and good interfacial bonding with Ni substrates. The interfacial hardness was equal to that of the sintered alloys and Ni substrates. Loose nickel powders ensured the density and interfacial bonding of the sintered alloys, the infiltration sintering process can be simplified and easily applied to practice.

1. Introduction
Nickel-based superalloys are widely used in modern aero engines, gas turbines and the hottest components of thermal power plants, such as working blades, turbine discs, combustion chambers, etc. [1]. After long-term use, the corrosion pits, cracks and other defects will appear on the hot components. The high cost of replacing these components promoted people to develop repair methods to extend the life of blades [2-4].

In 1990s, Liburdi proposed a powder metallurgy repair process called Liburdi Powder Metallurgy (LPM), the blades could be repaired through the infiltration of low-melting-point liquid alloy into high-melting-point porous skeleton [2]. Although various welding processes such as laser cladding techniques have been applied for blade repair [3,4], the LPM repair process can repair larger gaps with stable properties. If the alloy powders have the same composition as the matrix, high sintering temperature is required, which will degrade the microstructures and properties of the matrix. It is known that B and Si can effectively reduce the liquidus temperature of nickel alloy [5], which provide a possibility that the powder metallurgical repair process can be applied at lower temperatures that can reduce the influence of sintering temperature on the matrix.

In our previous work, the Ni-B-Si alloy powders with low melting point were prepared by mechanical alloying, and the initial melting temperature of Ni-4B-4Si(wt.%) alloy powders could be reduced to 1038 °C [6]. In this paper, the infiltration sintering properties of the Ni-4B-4Si alloy powders are investigated, the effects of sintering temperature and nickel skeleton on the microstructures and interfacial bonding properties of sintered alloys are analyzed.
2. Experimental
Carbonyl nickel powders (99%, 10 μm), silicon powders (99.9%, 3 μm) and boron powders (99.9%, 15 μm) were used as the source powders. The Ni-4B-4Si (wt.%) alloy powders were prepared by ball milling for 30 h on a planetary ball mill at a rotation speed of 380 rpm. The source powders, WC balls and dispersing liquid (ethanol) were sealed in a stainless steel jar with ball-to-powder weight ratio of 10:1. The preparation procedure and characterizations of Ni-4B-4Si alloy powders were reported in detail previously [6].

The as-prepared Ni-B-Si alloy powders were cold pressed and placed on the loose nickel powders (nickel powders on Ni substrate) and compact nickel powders (after cold press), respectively. The infiltration sintering was then performed at 1050-1150 °C for 2 h.

The phase composition and microstructures of the sintered alloys were characterized by 7000S X-ray diffraction (XRD) and JSM-6700F scanning electron microscopy (SEM), respectively. The hardness near the interfacial regions between Ni substrates and sintered alloys was tested by Aglient-G200 nanoindentation.

3. Results and discussion
Figure 1(a-c) and (d-f) shows the SEM images of infiltration regions of sintered alloys prepared using loose and compact nickel powders, respectively. When the sintering temperature was 1050 °C, sintering necks were formed between the nickel powders during the sintering process, meanwhile, the Ni-B-Si alloy powders were melted and infiltrated into the as-formed nickel skeleton. However, the poor fluidity of the Ni-B-Si liquid alloy due to the low sintering temperature resulted in few Ni-B-Si alloy phases in the infiltration region. With the increase of sintering temperature, the fluidity of the liquid alloy was improved, the content of Ni-B-Si alloy phases in the infiltration region was obviously increased and the microstructures became dense.

![SEM images of infiltration regions of sintered alloys prepared using (a-c) loose and (d-f) compact nickel powders at temperatures of (a,d) 1050 °C, (b,e) 1100 °C, (c,f) 1150 °C.](image)

As shown in figure 1(a-c), when the Ni-B-Si liquid alloy was infiltrated into the loose nickel powders, the capillary force and viscous flow of the liquid alloy between the nickel powders promoted the continuous infiltration of liquid alloy into the nickel skeleton. During the sintering process, sintering necks were formed between the nickel powders, the Ni-B-Si liquid alloy filled in the gaps of nickel skeletons and the compact sintered alloys were formed by metallurgical bonding. As shown in
figure 1(d-f), the nickel powders deformed and became compact after cold press, which facilitated the formation of sintering necks between nickel powders during sintering process. During the sintering process, the elastic recovery of nickel powders occurred and a wide range of sintering necks were formed, which blocked the further infiltration of liquid alloy into the nickel skeleton, resulting in few Ni-B-Si alloy phases in the infiltration region.

Figure 2(a-c) and (d-f) shows the SEM images of interface regions between Ni substrates and sintered alloys prepared using loose and compact nickel powders, respectively. As shown in figure 2(a-c), after the infiltration of Ni-B-Si liquid alloy into the loose nickel powders, the interface between Ni substrate and sintered alloy exhibits good metallurgical bonding. When the sintering temperature increased to 1150 °C, the fluidity of the Ni-B-Si liquid alloy was enhanced, a large amount of Ni-B-Si alloy phases were formed near the interface, which weakened the interfacial bonding between Ni substrate and sintered alloy.

![Figure 2. SEM images of interface regions between Ni substrates and sintered alloys prepared using (a-c) loose and (d-f) compact nickel powders at temperatures of (a,d) 1050 °C, (b,e) 1100 °C, (c,f) 1150 °C.](image)

As shown in figure 2(d-f), the Ni-B-Si liquid alloy was infiltrated into the compact nickel powders. When the sintering temperature was 1050-1100 °C, the liquid alloy did not infiltrate to the interface, near which a large number of pores and sinters formed by the compact nickel powders are observed. During the sintering process, a lot of sintering necks were formed among the compact nickel powders, the gaps of the nickel skeleton became narrow, which was not helpful to the infiltration of Ni-B-Si liquid alloy. The fluidity of the liquid alloy was quite poor at low sintering temperature and the infiltration distance was limited. The increase of sintering temperature to 1150 °C facilitated the infiltration of Ni-B-Si liquid alloy, the pores near the interface disappeared and the compactness was obviously improved.

Figure 3 shows the hardness distribution near the interface regions between Ni substrates and sintered alloys prepared using loose and compact nickel powders at a sintering temperature of 1100 °C. After the infiltration of Ni-B-Si liquid alloy into the loose nickel powders, the interfacial hardness is equal to that of Ni substrate and sintered alloy, which reveals the good bonding property between Ni substrate and sintered alloy. As shown in figure 2(b), the Ni-B-Si liquid alloy infiltrated to the Ni substrate, enhancing the interfacial bonding of the sintered alloy and Ni substrate. After the infiltration of Ni-B-Si liquid alloy into the compact nickel powders, the interfacial hardness decreased sharply. As
shown in figure 2(e), the Ni-B-Si liquid alloy did not infiltrate to the Ni substrate, the large number of pores distributing near the interface weakened the interfacial bonding between the sintered alloy and Ni substrate.

**Figure 3.** Hardness distribution near interface regions between Ni substrates and sintered alloys prepared using loose and compact nickel powders at a sintering temperature of 1100 °C.

**Figure 4.** XRD pattern of Ni-B-Si alloy sintered at 1100 °C.

Figure 4 shows the XRD pattern of Ni-B-Si alloy sintered at 1100 °C. Ni-B-Si sintered alloy mainly consists of γ-Ni and Ni3B. In combination of the microstructures of the sintered alloys shown in figures 1 and 2, it can be seen that the light phase is γ-Ni and the dark phase is Ni3B. By comparing the Ni-B and Ni-Si phase diagrams [5], it can be seen that the solid solubility of B in Ni (0.3 at.%) at room temperature is much lower than that of Si in Ni (8 at.%). Meanwhile, the diffusion coefficient of B in Ni (6.22×10^{-11} m^2 s^{-1}) at 1100 °C is much higher than that of Si in Ni (3.09×10^{14} m^2 s^{-1}) [7]. Therefore, B easily diffused from the melt and precipitated in the form of Ni3B [8]. When the melt temperature decreased to the eutectic temperature, γ-Ni and Ni3B were formed by eutectic reaction, and Si mainly solid soluted in the γ-Ni. The formation of Ni3B can improve the hardness of sintered alloys [9, 10], which contributed to the high interfacial hardness as shown in figure 3.

4. Conclusions
Ni-4B-4Si (wt.%) alloy powders were infiltrated into the nickel skeletons. γ-Ni and Ni3B were formed in the infiltration regions and Si solid soluted in the γ-Ni. The loose nickel powders facilitated the infiltration of Ni-B-Si liquid alloy into the nickel skeleton as compared to the compact nickel powders, the sintered alloys became more compact and had better interfacial bonding with Ni substrate. After infiltration of Ni-B-Si liquid alloy into the loose nickel powders, the interfacial hardness was equal to that of Ni substrate and sintered alloy, which was higher than that using compact nickel powders.

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**References**
[1] García Navas V, Arriola I, Gonzalo O and Leunda J 2013 *International Journal of Machine Tools and Manufacture* 74 19
[2] Ellison K A, Lowden P and Liburdi J 1994 *Journal of Engineering for Gas Turbines and Power* 116 237
[3] Sexton L, Lavin S, Byrne G and Kennedy A 2002 *Journal of Materials Processing Technology* 122 63
[4] Shepeleva L, Medres B, Kaplan W D, Bamberger M and Weisheit A 2000 *Surface and Coatings Technology* **125** 45
[5] Okamoto H, 2000 *Desk Handbook: Phase Diagrams for Binary Alloys* ASM International, Materials Park, OH
[6] Wang F, Yang Q, Zou J and Liang S 2015 *Rare Metal Materials and Engineering* **44** 1985
[7] Pouranvari M, Ekrami A and Kokabi A H 2013 *Journal of Alloys and Compounds* **563** 143
[8] Binesh B and Gharchbagh A J 016 *Journal of Materials Science and Technology* **32** 1137
[9] Lopez J R, Mendez P F, Perez-Bueno J J, Trejo G, Stremsdoerfer G and Meas Y 2016 *International Journal of Electrochemical Science* **11** 4231
[10] Yuan X, Kang C Y and Kim M B 2009 *Materials Characterization* **60** 923