



## Article

# The Influence of Ni-Added Fe-Based Pre-Alloy on Microstructure Evolution and Lifetime Extension of Diamond Tools

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**Abstract:** Diamond tools were prepared by sintering Fe-Cu-Sn-Zn-Ni pre-alloyed powders and diamonds. The effects of Ni contents in pre-alloyed powders on microstructure evolution of Fe-based matrix, the properties of Fe-based matrix and the service life of diamond tools were investigated. The results showed that adding 3~15 wt.% Ni into the Fe-Cu-Sn-Zn pre-alloyed powders refined the microstructure of the Fe-based matrix and improved its density and hardness gradually. The addition of Ni reduced the loss of low melting liquid phase at a low sintered temperature, thus resulting in a decrease of the pores, an increase of the density and hardness of Fe-based matrix. When the Ni content is less than 9 wt.%, the bending strength of Fe-based matrix and diamond tools, together with the holding force of Fe-based matrix to diamonds increases sharply. They reached up to the optimal value with the Ni content of 9 wt.%. At this sintering powder ratio, the sufficient Fe-Cu-Sn-Zn-Ni liquid phase had a good wettability on the surface of diamonds, thus the optimal performance of sintered matrix and diamond tools was obtained. The service life of diamond tools was prolonged greatly owing to the excellent bonding capacity between matrix and diamonds. Once the Ni content exceeded 9 wt.%, the corresponding value decreased gradually. The fracture morphologies of the matrix changed from the brittle fracture into brittle-ductile fracture, then ductile fracture (with the Ni content of 9 wt.%), brittle-ductile mixed fracture and brittle fracture.

**Keywords:** Fe-Cu-Sn-Zn-Ni pre-alloyed powders; diamond tools; density; bending strength; holding ability



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## 1. Introduction

Diamond tools are usually sintered by diamond particles and metal-based powders through powder metallurgy technology such as hot pressing or cold pressing-sintering [1]. In order to maintain the sharpness of diamond tools and prolong their service life during cutting or polishing, the holding force of the matrix to diamonds is very important. It is generally known that Co-based matrix has a strong holding ability to diamonds, which has excellent comprehensive properties, such as good red hardness, toughness and self-sharpening. However, its price is high and its reserves are short, the application of Co-based matrix is limited [2–4]. Therefore, researchers are committed to the development of Co free or low Co metal matrix.

Fe is rich in resources and is low in price. It has many similar properties with Co. Moreover, it has a good wettability on the surface of diamonds, which can produce a good bonding force between Fe-based matrix and diamonds, finally, their interfacial bonding

strength is improved [5,6]. Thus Fe has become a hot spot in the research of sintered matrix materials in recent years [7,8].

In general, Fe is susceptible to eroding the diamond seriously at a high sintered temperature (900~1100 °C), thus the diamond is easy to be graphitized, which results in a decrease in the strength and the service life of diamond tools. In this case, it is necessary to decrease the sintered temperature of Fe-based metal matrix in order to improve its comprehensive properties and expand its applications. Some research results [9–12] show that using pre-alloyed powders to replace powders particles in a sintered matrix can decrease the sintered temperature. Chen Zhengwei et al. [13] studied the application of Fe-based pre-alloyed powders in diamond tools. He concluded that the properties of Fe-based matrix material can reach the level of Co-based matrix material as long as the formulation of Fe-based pre-alloyed powders was reasonable. In addition, some scholars insist on one view that adding Cu, Sn and Zn into Fe powders can effectively decrease the sintered temperature of Fe-based matrix and ensure its good comprehensive performance [14–17]. However, the melting point of Sn and Zn is low, so the loss of Fe-Cu-Sn-Zn liquid phase is serious at a low sintered temperature, which weakens the binder action of Fe-Cu-Sn-Zn liquid phase.

Ni has a high melting point and density, and it is a favorable strengthening and toughening element for Fe-based powder metallurgical materials [18,19]. Therefore, in this paper, different Ni contents are added into Fe-Cu-Zn-Sn powders, and then they are prepared into pre-alloyed powders. These pre-alloyed powders and diamonds will be sintered into diamond tools by powder metallurgy. The effects of different Ni contents in pre-alloyed powders on the properties of Fe-based sintered matrix and diamond tools will be discussed. The influence mechanism of Ni-containing pre-alloyed powders on the life extension of diamond tools will be analyzed. These studies aim to enrich the theoretical basis for the preparation of diamond tools.

## 2. Materials and Methods

### 2.1. Experimental Materials

The raw materials included iron powder, copper powder, tin powder, nickel powder, zinc powder and self-made pre-alloyed powder. The particle size was 200 mesh. The pre-alloyed powder was prepared by multistage tightly coupled atomization. The composition ratio of pre-alloyed powder was listed in Table 1. The diamond with the brand of HTD2620 was chosen. Its size was 40 mesh. The concentration of diamond was 20% (based on 400% concentration system). In order to study the effect of Ni content in the pre-alloyed powder on the microstructure of Fe-based matrix and the performance of matrix and cutter head of diamond tools, six kinds of composition design were formulated, which were marked as T1, T2, T3, T4, T5 and T6, with Ni contents of 0%, 3%, 6%, 9%, 12% and 15%, respectively.

**Table 1.** The composition ratio of pre-alloyed powder.

Composition	Ratio (wt.%)
Ni	0–15
Sn	2–10
Cu	15–25
Zn	10–15
Fe	Balance

### 2.2. Experimental Process

The powders were weighted according to the composition design shown in Table 1. The powders were mechanically mixed in a three-dimensional mixer for 30 min. In the mixing process, about 2% paraffin or kerosene was added to prevent the segregation of the powders and facilitate the press molding of the cutter head. Then, they were put into the Graphite Mold to be sintered in the hot press sintering machine (SMVB60). During the sintering process, the sintering parameters were the highest temperature of 800 °C and the

holding time of 60 s. After sintering, the size of samples was 40 mm × 8 mm × 3.2 mm. The specific sintering process was shown in Figure 1.

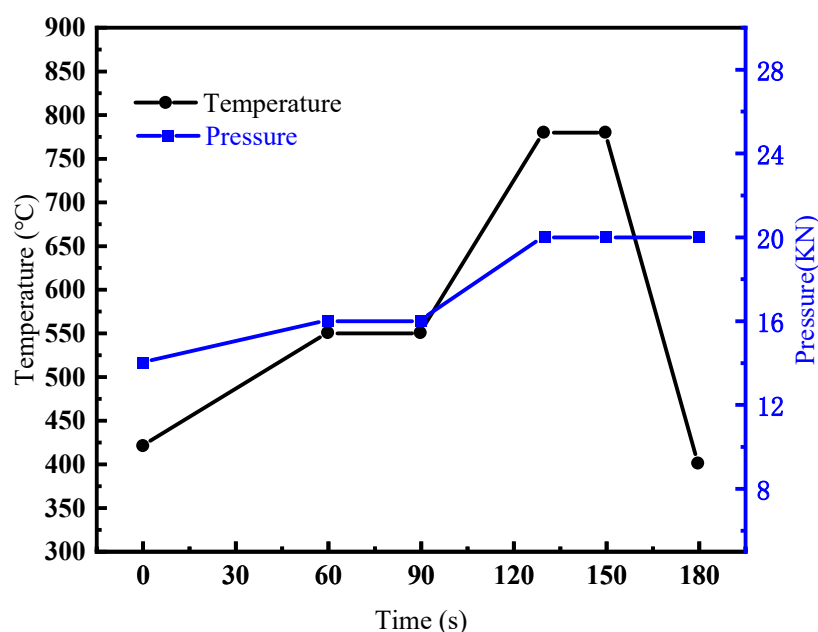


Figure 1. Sintering process of powders.

There are some details to pay attention to during the experiment. Cu and Ni in the powders are easily oxidized at room temperature. Due to the existence of oxides, on the one hand, the graphitization of diamond will be intensified and the surface of the diamond will be strongly eroded, finally, the cutting performance of diamond will be reduced. On the other hand, pores may be formed in the sintering process, which results in the reduction of the strength of the matrix and cutter head. Therefore, the powder storage equipment includes gas-making equipment and a reduction furnace. The reducing agent is hydrogen or coal gas. Moreover, graphite mold is selected as a carrier during powder sintering. The compressive strength of graphite shall not be less than 40 MPa, the porosity of graphite shall not be greater than 30%, and the density shall be above 1.6 g/cm<sup>3</sup>.

### 2.3. Performance Testing and Microstructural Analysis

The immersion paraffin method was used to measure the sintering density of samples according to the standards GB/T10451-2002 and GB 3850-83. The sample after being immersed in paraffin at the temperature of 170 °C was weighed in the air and in the water, respectively. The sintering density of samples was calculated by the formulation of  $\rho = m_{air} \cdot \rho_{water} / (m_{p-air} - m_{p-water})$ , where  $m_{air}$  is the weight of the samples in the air,  $m_{p-air}$  is the weight of the samples that are immersed in paraffin in the air,  $m_{p-water}$  is the weight of the samples that are immersed in paraffin in the water and  $\rho_{water}$  is the density of the water. The theoretical density of samples was calculated according to the formula  $\rho_0 = 100 / (\frac{m_1}{\rho_1} + \frac{m_2}{\rho_2} + \frac{m_3}{\rho_3} + \dots + \frac{m_n}{\rho_n})$ , where  $m_1, m_2 \dots m_n$  represents the mass percentage of each component in the sample and  $\rho_1, \rho_2 \dots \rho_n$  is the theoretical density of each component in the sample. The sample density was calculated by the formulation of  $\eta = \rho / \rho_0$ .

The hardness of sintered matrixes was measured perpendicular to the pressing direction by a Rockwell hardness tester (HRB). The reported values are an average of five data points.

Three-point bending tests were carried out to measure the bending strength of the sintered matrix and diamond tools on a universal testing machine (CMT5205) under the

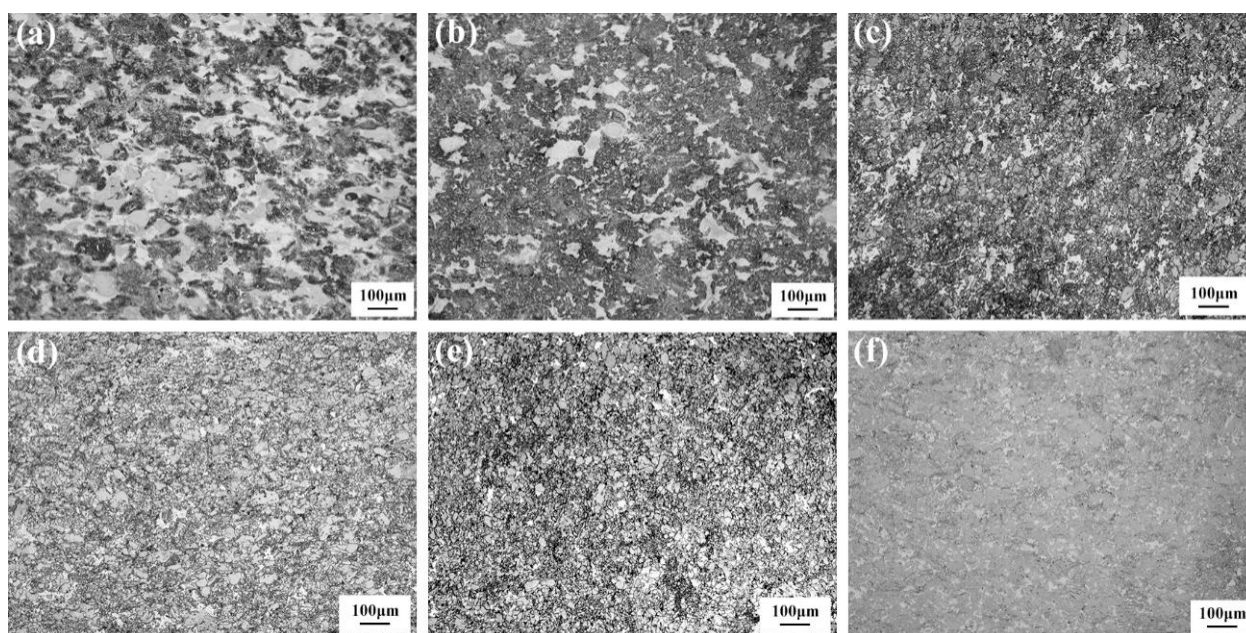
loading speed of 1mm/min and span length of 30 mm. The bending test was repeated 5 times and the final result was taken as the average value.

The microstructure of the sintered matrix was observed by a ZEISS optical microscope (OM, Oberkochen, Germany). The fracture morphologies of the sintered matrix and diamond tools were analyzed by a JSM-6480 scanning electron microscope (SEM, JEOL, Tokyo, Japan). The chemical composition of the fracture was tested by Energy Dispersive Spectrometer (EDS). The tissue of sintered matrix was analyzed by an XRD-6000 X-ray diffractometer instrument (XRD, Shimadzu, Kyoto, Japan). X-ray diffraction (XRD) analysis was carried out with Cu-K $\alpha$  radiation and scanning angles ( $2\theta$ ) between  $10^\circ$  and  $90^\circ$ .

### 3. Results and Discussion

#### 3.1. Effect of Ni Content on the Microstructure

Figure 2 shows the microstructure of sintered matrixes with different Ni contents. When the pre-alloyed powders contain Ni-free, the grain size of sintered matrix is coarsened, some light-colored copper-based bonding phase segregates at the grain boundary, some continuous “linear” pores and a few irregular pores appear, as shown in Figure 2a. Figure 2b–f show that the grains gradually become fine and those light-colored copper-based bonding phase begin to distribute uniformly in the sintered matrix, and those continuous “linear” pores and irregular pores disappear gradually. It can be concluded that the addition of Ni in pre-alloyed powders plays the role of grain refinement on the sintered matrix and it can reduce the number of pores of the sintered samples. Moreover, the grain size grows finer with the increase of the Ni content.



**Figure 2.** Microstructure of sintered matrixes with different Ni content. (a) Ni-free; (b) with 3. wt.% Ni; (c) with 6 wt.% Ni; (d) with 9 wt.% Ni; (e) with 12 wt.% Ni; (f) with 15 wt.% Ni.

Figure 3 shows the variation of the sintering density  $\rho$ , the theoretical density  $\rho_0$  and the density  $\eta$  of sintered matrix under different Ni contents in pre-alloyed powders. As the addition of Ni in pre-alloyed powders is less, the theoretical density  $\rho_0$  of sintered matrix changes little. The sintering density  $\rho$  as well as the density  $\eta$  increase gradually and the porosity decreases with the increase of Ni content in the pre-alloyed powder. As Sn and Zn in the powder have low melting points, they can form a liquid phase at low sintering temperature. Then these liquid phase fills the voids in the sintered matrix so that the density of the matrix is improved. However, the liquid phase with a low melting point is easy to flow away at lower sintering temperatures, so the role of Cu, Zn and Sn



as bonding phase is weakened. Due to the high melting point of Ni, the addition of Ni reduces the loss of low melting point liquid phase. Therefore, the sintering density  $\rho$  as well as the density  $\eta$  increases with the increase of Ni contents.

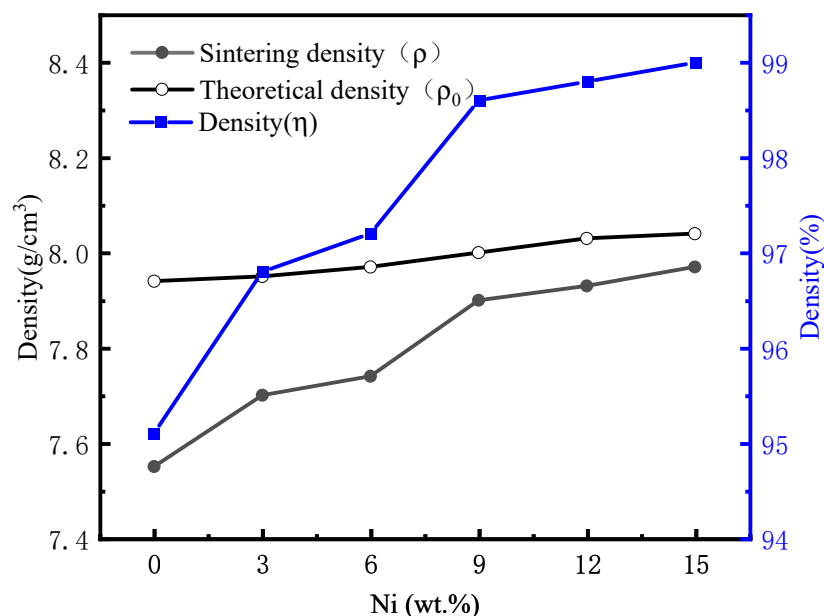


Figure 3. Density of the sintered matrix with different Ni contents.

Figure 4 shows the XRD analysis results of the matrix phase with different Ni content. The matrix phase is composed with  $\text{Cu}_3\text{Sn}$ ,  $\text{Cu}_5\text{Zn}_8$  and Fe as Ni is free in the pre-alloy (see Figure 4a). When Ni is added into the pre-alloy, the new phase of Fe-Ni solid solution are observed, as shown in Figure 4b. With the addition of Ni, Fe-Ni solid solution is formed in the microstructure, which refines the matrix microstructure and improves the matrix density.

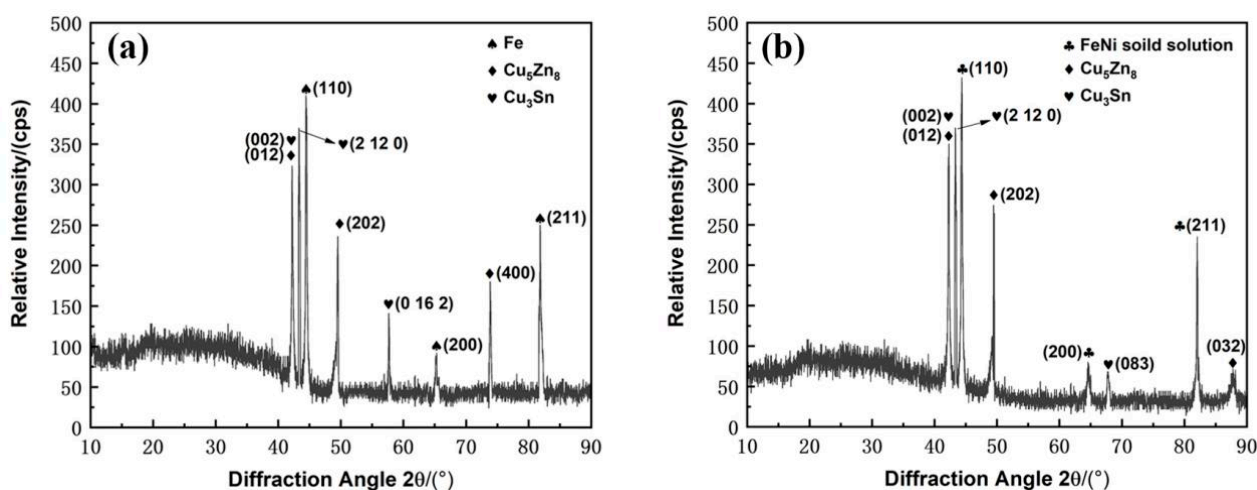


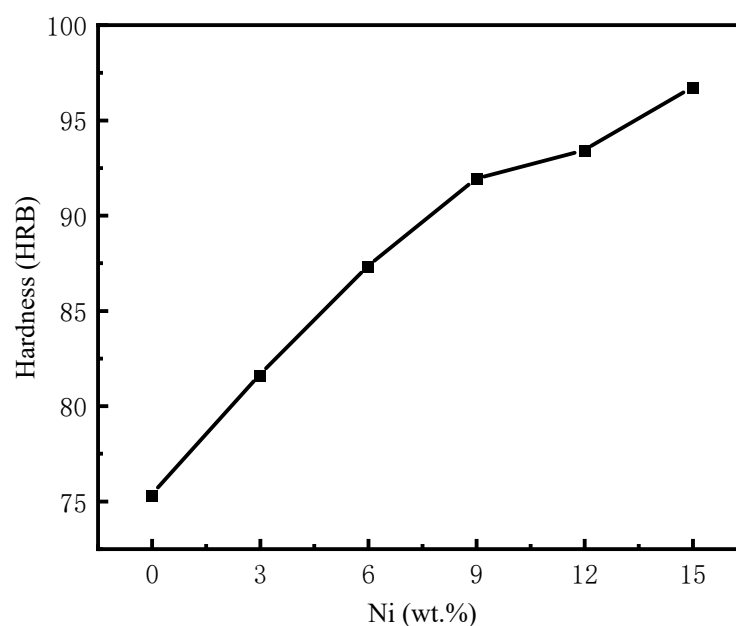
Figure 4. XRD analysis results of the matrix phase: (a) Ni-free; (b) with 15 wt.% Ni.

### 3.2. Lifetime Extension of Ni-Added Fe-Based Pre-Alloy Brazing Coating in Diamond Tools

#### 3.2.1. Effect of Ni Content on Hardness of Matrix

Figure 5 shows that the Rockwell hardness of sintered matrix with different Ni contents in powders. When the powder does not contain Ni, the hardness is only 75.3 HRB. When the Ni content is increased from 0% to 15%, the hardness is increased from 75.3 HRB to 96.7 HRB. The results are explained as follows. Firstly, the sintered matrix consists of

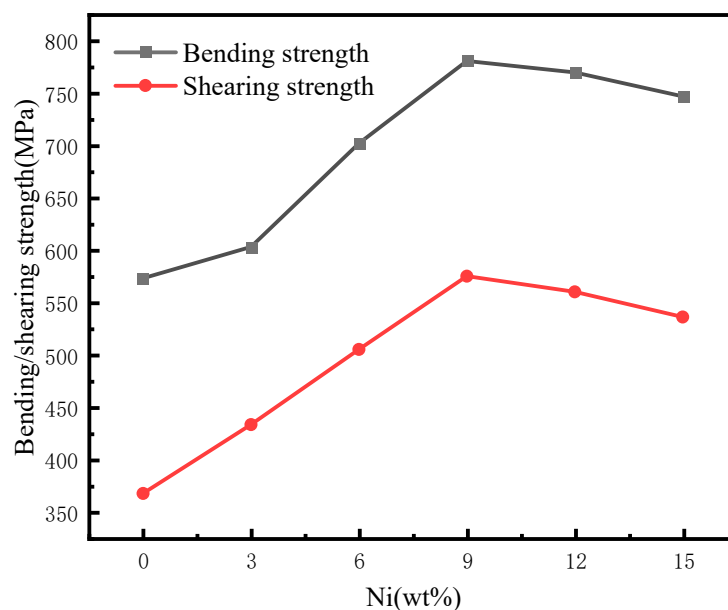
framework materials, pre-alloyed powders and diamonds. Diamonds are brazed in the framework materials using the pre-alloyed powder as a bonding phase. The pre-alloyed powder has higher hardness than the framework materials due to its preparation process. With the increase of Ni content, the proportion of pre-alloyed powder increased, so the hardness of sintered matrix increased. Secondly, with the increase of Ni, the loss of liquid phase produced by low melting point material is reduced and more liquid phase fills the gaps among the powders, thus the density and hardness increase.



**Figure 5.** The hardness of the matrix of sintered samples with different Ni contents.

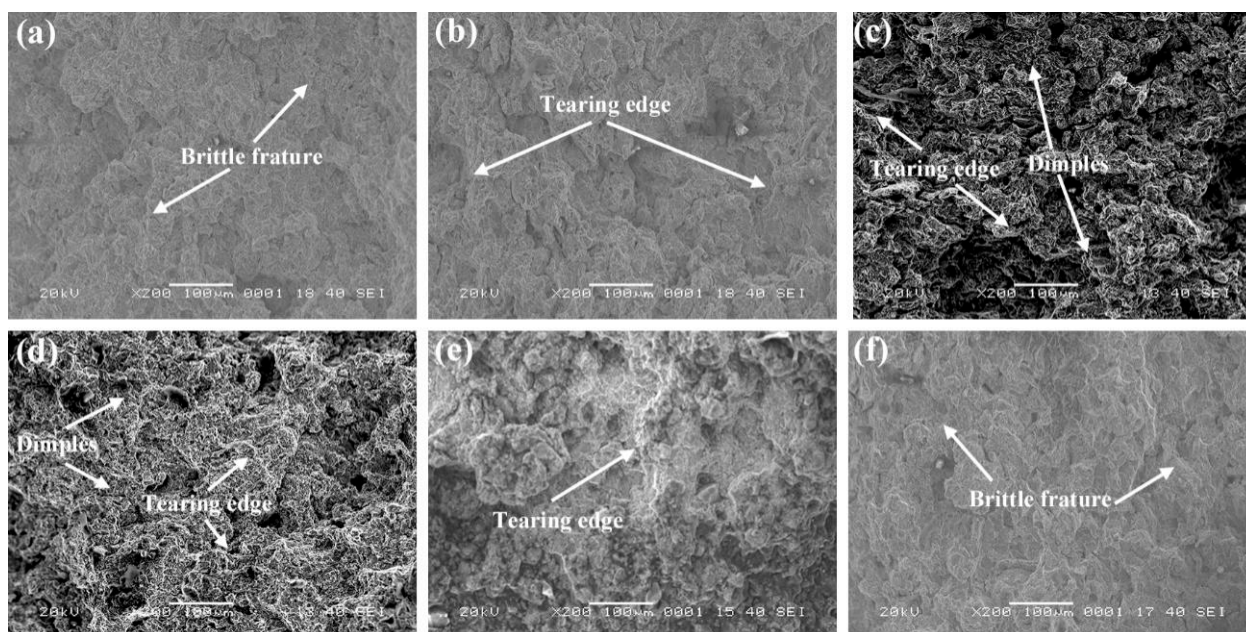
### 3.2.2. Effect of Ni Content on Shearing Strength and Bending Strength of Matrix

Figure 6 shows the bending strength and shearing strength of sintered matrixes with different Ni contents. The bending strength and shearing strength is 367 MPa and 573 MPa without Ni in the powders. With the increase of Ni, the bending strength and shearing strength increases. When the Ni content is from 3 to 9 wt.%, the bending strength and shearing strength increase rapidly. While the Ni content exceeds 9 wt.% and increased to 15 wt.%, the bending strength and shearing strength decreases slightly. Some paper note that the hardness, bending strength and shearing strength is closely relevant to the density for powder metallurgy, meanwhile, these values increase with the increase of density [20]. When the Ni content increases, the reduction of liquid phase loss leads to more liquid phase filling the gap between the powder, thus the hardness increase and bending strength together with shearing strength also increase. Moreover, Figure 2 shows the grain size becomes smaller with the increase of Ni content and compounds of  $\text{Cu}_3\text{Sn}$  phase and  $\text{Cu}_5\text{Zn}_8$  phase are gradually fine and dispersed. It can infer the mechanical properties increase, so the bending strength and shearing strength increase. However, the Ni content exceeds 9 wt.%, the melting point of Fe-Cu-Sn-Ni-Zn is improved and the loss of liquid phase is decreased, thus the excessive  $\text{Cu}_3\text{Sn}$  phase forms, and the bending strength and shearing strength of the sintered matrix decreases slightly.



**Figure 6.** Bending strength and shearing strength of sintered matrix with different Ni contents.

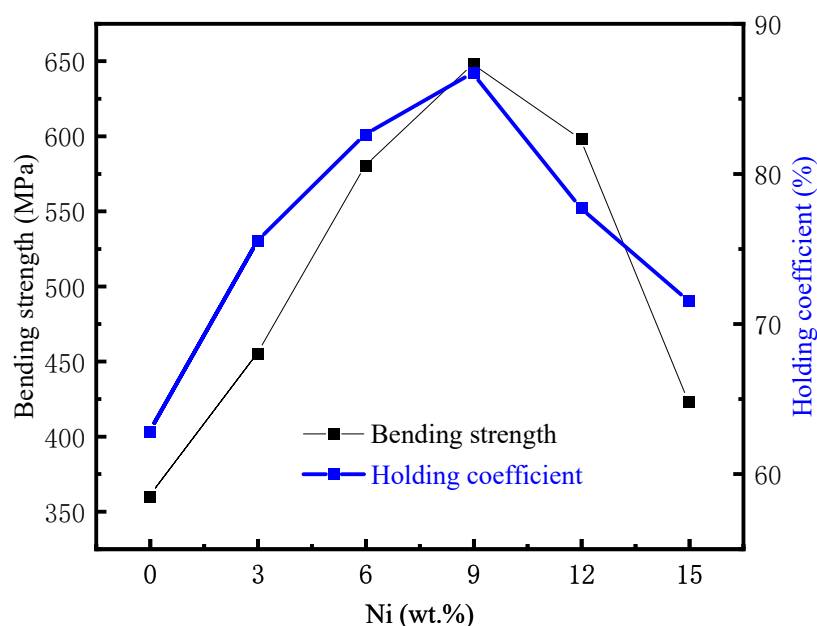
Figure 7 shows the SEM fracture morphology of the sample after three-point bending under different Ni contents. The Ni-free sample shows a brittle-ductile mixed fracture, and some dimples exist at the fracture, while the particles in the fracture are bigger and the number of pores is more. With the Ni content increased up to 9 wt.%, the fracture mode is ductile and there are more dimples at the fracture of samples. The uneven fracture surface indicates that these samples experience large plastic deformation during the bending process. However, the Ni content exceeds 9 wt.%, the fracture shows mainly brittle fracture. There are obvious cleavage steps at the fracture.



**Figure 7.** Fracture morphologies of the sintered matrixes with different Ni contents: (a) Ni-free, (b) with 3 wt.% Ni, (c) with 6 wt.% Ni, (d) with 9 wt.% Ni, (e) with 12 wt.% Ni, (f) with 15 wt.% Ni.

### 3.2.3. Influence of Ni on Bending Strength of Cutter Head of Diamond Tools

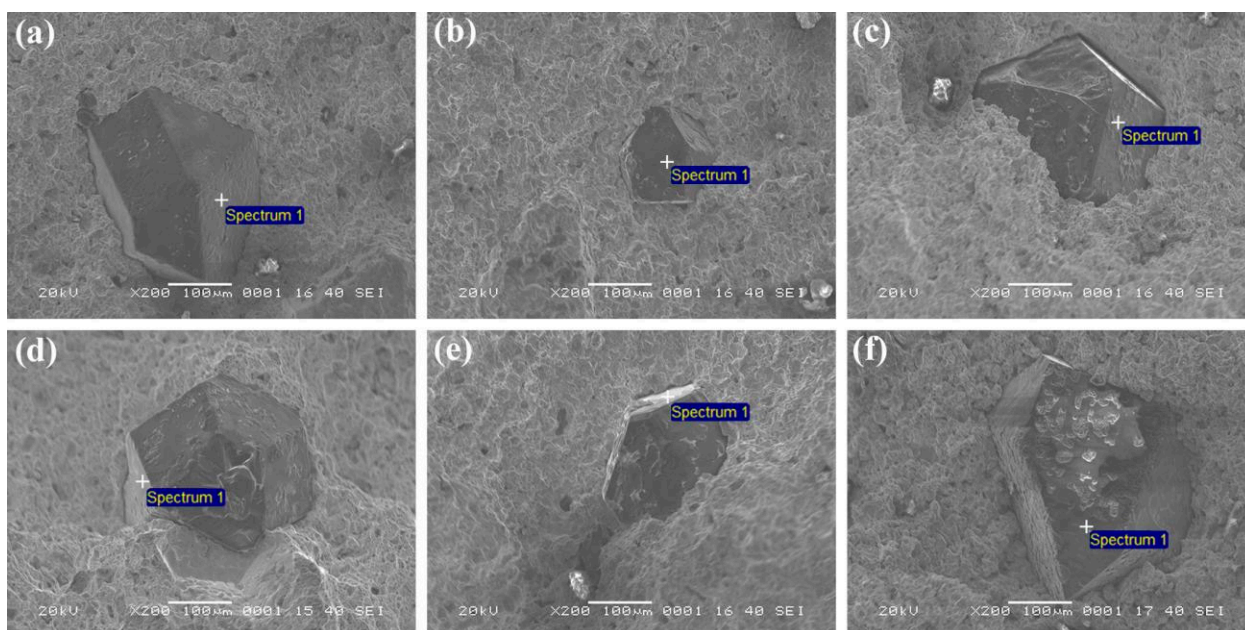
The cutter head is made by adding diamond into the mixture powder through the process of mixing, pressing and sintering. Figure 8 shows the bending strength of diamond tools and the holding coefficient of the sintered matrix to diamond under different Ni contents in pre-alloyed powder. The bending strength increases first, then decreases, and reaches the maximum with the Ni content of 9 wt.%. Moreover, Ni has a significant effect on the holding coefficient of sintered matrix to diamond. The holding coefficient represents the acting force of sintered matrix to diamond for the diamond cutter head [18]. The holding coefficient is consistent with the bending strength of the cutter head. The value of holding coefficient is up to the maximum when the Ni content is 9 wt.%. This indicates it has a good holding force of sintered matrix to diamond with the Ni content of 9 wt.%.



**Figure 8.** The effect of Ni content on the bending strength of diamond tools and holding coefficient of the sintered matrix on diamond.

Figure 9 shows the bending fracture morphologies of diamond tools under pre-alloyed powders with different Ni contents. It can be seen from Figure 9a that the fracture of diamond tools contains continuous linear pores with Ni-free in the pre-alloyed powders, which corresponded to a low holding coefficient of the sintered matrix to the diamond and a low bending strength of diamond tools. When the Ni content of pre-alloyed powders increases from 3 to 9 wt.%, the number of linear pores in the fracture of diamond tools decreases, and the structure distribution of the matrix is uniform, thus improving the density of the sintered matrix, enhancing the holding ability of the sintered matrix to the diamond and increasing the bending strength of diamond tools (see Figure 9b–d). The lifetime extension of the diamond tool reaches the optimal value with the Ni content of 9 wt.%. When the Ni content exceeds 9 wt.%, there is some crack at the fracture and the corresponding service life of diamond tools is decreased (see Figure 9e,f).





**Figure 9.** The bending fracture morphologies of diamond tools under pre-alloyed powders with different Ni contents: (a) T1: Ni-free, (b) T2: with 3 wt.% Ni, (c) T3: with 6 wt.% Ni, (d) T4: with 9 wt.% Ni, (e) T5: with 12 wt.% Ni, (f) T6: with 15 wt.% Ni.

The EDS results of the interface between the matrix and the diamond are listed in Table 2. It can indicate that metallurgical reactions take place between the matrix and the diamond for all samples with different content of Ni. It is well known that Fe has a good wettability on the surface of the diamond. It diffuses to and reacts with diamonds to form carbides, which improve the holding ability of matrix to the diamond. When the Ni content in pre-alloyed powders is less than 9 wt.%, Ni has good solubility with Cu and Fe, under the action of solid solution strengthening and dispersion strengthening among them, the microstructure of matrix can be refined, therefore the strength and toughness of matrix is improved. Thus, the service life of diamond tools is enhanced with the increase of Ni content. The holding ability of the matrix to the diamond reaches the optimum value with the Ni content of 9 wt.%. However, when the Ni content increases from 9 to 15 wt.%, the excessive Ni is added into the liquid phase to improve the melting point of Fe-Cu-Sn-Ni-Zn so that the Cu-Sn brittle phase is formed. Thus, the toughness of the matrix decreases, which leads to the decrease of the bending strength of diamond tools and the slight deterioration of the holding ability of the matrix to diamond, and finally the service life of diamond tools is decreased.

**Table 2.** EDS results of the interface between the matrix and the diamond.

Ni Content (wt.%)	Chemical Elements/at.%					
	C	Fe	Cu	Sn	Ni	Zn
0	98.07	1.12	0.50	0.12	-	0.16
3	96.12	1.15	0.63	2.03	0.07	0
6	96.05	2.53	0.94	0	0.29	0.18
9	80.69	13.72	3.46	0.99	0.45	0.69
12	98.59	1.27	0.09	0.01	0.01	0.04
15	96.34	1.21	1.32	0.55	0.47	0.11

#### 4. Conclusions

This present work studied the microstructure evolution of Ni-added Fe-based sintered matrix and the lifetime extension of Ni-added diamond tools. The main conclusions were as follows:

- With the increase of Ni content in the pre-alloyed powders, the microstructure of the matrix is refined and becomes more uniform; the hardness of the matrix increases.
- When the content of Ni increased from 0 to 15 wt.%, the bending strength and the shearing strength both increased firstly, then reached the optimal value with the Ni content of 9 wt.% and decreased finally. The fracture morphologies of the matrix changed from brittle fracture into brittle-ductile fracture, then ductile fracture (with the Ni content of 9 wt.%), brittle-ductile mixed fracture and brittle fracture.
- With the increase of Ni content in the pre-alloyed powders, the bending strength of diamond tools and the holding coefficient of the sintered matrix to diamond increased firstly, then reached up to the maximum with the Ni content of 9 wt.% and decreased finally.
- The service life of diamond tools was greatly prolonged when adding the Ni content of 9 wt.% into the pre-alloyed powder.

**Author Contributions:** Conceptualization: J.P., Y.S., W.L. and M.W.; methodology: D.L. and J.P.; formal analysis: J.P. and S.Z.; resources: S.X.; data curation: S.X. and S.Z.; writing—original draft preparation: J.P.; writing—review and editing: J.P. and W.L.; writing—manuscript finalization: J.P., W.L. and D.L.; supervision: S.X. and W.L.; project administration: W.L. and S.Z.; funding acquisition: S.Z. and S.X. All authors have read and agreed to the published version of the manuscript.

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