Fe-based sintered matrix for diamond tools: microstructure, mechanical performance and crack initiation and propagation characteristics

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Abstract
To design Fe-Cu-Sn-Ni metal binders for diamond tools and optimize the performance of binders, Fe-based binders were prepared by hot-press sintering method using Fe powder, Cu powder, Sn powder and Ni powder as the raw materials. The phase constitution, microstructure, mechanical properties and crack formation were evaluated. Results showed that Fe-based matrixes are composed of Cu3Sn intermetallic compounds and several solid solutions, such as α-Fe, γ(Fe,Ni), and Cu13.7Sn. The relative high Sn content can increase the hardness of the sintered bulk samples and significantly reduce the bending strength. With the increase of Ni content, the hardness increases gradually, while the bending strength increases firstly and then decreases. The cracks initiate from brittle Cu3Sn intermetallic phases and quickly propagate in the brittle phases or along the interface between Cu3Sn intermetallic compounds and γ(Fe,Ni) phases. The propagation of crack can be impeded by Cu13.7Sn solid solution.

1. Introduction
Diamond tools are widely used in natural stone processing, ceramic modification, building materials, mechanical processing, geological drilling, mining industry and electronics industry [1, 2]. Because synthetic diamonds are mostly fine particles, the working pieces of diamond tool is a composite material of diamond single crystals and metal binder, and diamond particles are embedded in the metal binder matrices. It is the largest production of diamond tools, with about two-thirds of the entire diamond tool market [3].

Two main fabrication methods are commonly applied in the sintered diamond tool industry. Both the cold-press and the hot-press sintering are techniques for the mass fabrication of diamond sintered compacts. Cold-press sintering method contains two successive procedures, which are cold pressing and vacuum sintering. The compacts of diamond particles and metal binder are prepared by cold pressing method, and then can be sintered in a vacuum. Besides, continuous throughput of composite sintered compacts can be implemented by a conveyor furnace [4]. In contrast, the hot-press sintering technique is a combination of the heating process and the mechanical compression of the diamond-metal powders as one single step. Therefore the total production speed of hot-press sintering (from the powder mixture to the sintered compact) is obviously faster than that of the cold-press sintering. On the other hand, the sintered compact produced by hot-press sintering has relatively high density because of the synchronized pressing and heating [5]. Therefore, the hot-press sintering process is used in this work.

The metal binder generally containing multi-component metal, and the properties of diamond tools largely depend on the properties of metal binder [6, 7]. To obtain good mechanical properties and long service life of diamond tools, the properties of metal binder should possess a combination of excellent mechanical properties [8, 9]. Therefore, it is important to improve the properties of metal binders for diamond tools [10]. It is conventionally recognized that Co is the best matrix material for fabricating binders due to its excellent chemical
compatibility with diamond, matching wear resistance, and low sintering temperature. However, as an important strategic resource, the high cost restricts the application of Co in the diamond tools, especially in middle and low-end markets [3, 11]. Co-based metal binders have been replaced by multi-component alloys. Fe and Co are congeners which has similar properties, such as same crystal structures, good mechanical properties and better wettability to diamonds. However, Fe-based matrices need higher sintered temperature than Co-based matrix, so diamond is more prone to graphitization, resulting in the performance degradation of diamond composites. Besides, Fe-based matrices have some disadvantages such as low toughness and abrasive resistance [12, 13]. These reasons result that Fe-based diamond tools usually has poor working efficiency and short service life compared with Co-based diamond tools. Therefore, there were various studies carried out to improve the properties of Fe-based metal binders for diamond tools [14]. Some researchers focus on changing the manufacturing technique, such as using hot isostatic pressing sintering or microwave hot pressing [15]. Compared to the traditional sintering method, the microwave sintering displays relative low sintering temperature and much homogeneous element diffusion, high excellent holding force between in diamond and alloyed matrix. Novel perspective of the sintering process for the metallic matrix is provided. And the application of pre-alloying of Fe-based matrix powders can enhance the matrix’s strength, and reduce sintering temperature [16, 17]. Besides, some kind of nano-particles can be used to strengthen metal matrix. It has been found that the addition of trace amounts of nanoparticles can produce significant effects on strengthening the metal matrix [18, 19]. Mechanical performance of Fe-based Mn–Cu–Sn matrix is investigated, and the results show that Fe-Mn based matrix can replace Co-based alloys in some sintered diamond tools because of its excellent resistance to abrasion [20]. Based on the previous researches, the studies can be summed up as follows: process innovation, the pre-alloying of Fe-based matrix powders and the addition of strengthening components.

In this work, Fe-Cu-Sn-Ni sintered compacts were prepared by hot-press sintering which is widely used in the industry. The microstructure and properties of Fe-based metal binders were investigated, and the crack initiation and propagation of Fe-based sintered compacts were analyzed. We expect to obtain good microstructure and performance by regulating components. This approach has good economic value and can be applied in the actual production.

2. Materials and methods

2.1. Preparation of samples
The preliminary experiment showed that Cu element doesn’t obvious effect on the performance of metal binder. Based on the results of preliminary experiment, Cu content of metal binder is 25 wt%. The chemical composition of the metal binders is listed in table 1.

| Number | Fe  | Cu  | Sn  | Ni  |
|--------|-----|-----|-----|-----|
| A      | 62  | 25  | 10  | 3   |
| B      | 57  | 25  | 10  | 8   |
| C      | 59.5| 25  | 10  | 5.5 |
| D      | 64.5| 25  | 5   | 5.5 |
| E      | 54.5| 25  | 15  | 5.5 |

After mixing the metal powder, the mixtures were added into a graphite mould, and then were compacted into 40 × 8 × 3.5 mm³ samples by hot-press sintering. Hot-press sintering temperature was 1033K, sintering pressure was 18 MPa and sintering time was 4 min at the maximum temperature.

2.2. Investigation of microstructure
The phase component of the compacts were analyzed by X-ray diffraction (XRD) in the Bragg-Brentano geometry (monochromatic Cr-Kα radiation). The microstructure of sintered compacts was studied by Scanning Electron Microscope (SEM). X-ray energy dispersive spectrometry (EDS) equipped on SEM was also used to obtain the chemical analysis of the elements in selected points on the surface of compacts.

2.3. Test method of mechanical properties
The sintered compact samples were used to test the hardness and bending strength.

HR-150A Rockwell hardness tester was used to test Rockwell Hardness. Five points were tested on each sintered compacts, and four compacts were sintered for each metal binder. Therefore the value of Rockwell Hardness was the average value of twenty points.
The three-point bending strength of sintered compacts were carried out using MTS-C45.105 electronic universal testing machine equipped with an external digital controller. The fracture load of compacts was determined using the MTS TestSuite software. The span of supporting pins was 2.5 mm, the loading speed of crosshead was 1 mm min\(^{-1}\).

Bending strength was calculated by the equation (1). The value of bending strength was the average value of four sintered compacts.

\[
\sigma = \frac{3Fl}{2bh^2}
\]  

\(\sigma\), bending strength, MPa; \(F\), fracture load of bending, N; \(l\), the span, mm; \(b\), the width of sample, mm; \(h\), the height of the sample, mm.

3. Results and discussion

3.1. The microstructure of sintered compacts

Figure 1 shows the microstructure of the sintered compacts with different chemical composition. The EDS results of points in figure 1 are shown in table 2, which confirms that sintered compacts with different chemical

\begin{table}[h]
\centering
\begin{tabular}{cccc|cccc}
\hline
Point & Fe & Cu & Sn & Ni & Fe & Cu & Sn & Ni \\
\hline
1 & 93.73 & 2.79 & 0.40 & 3.09 & 92.80 & 3.14 & 0.84 & 3.22 \\
2 & 88.96 & 3.45 & 1.11 & 6.49 & 87.16 & 3.85 & 2.31 & 6.68 \\
3 & 5.15 & 83.43 & 10.60 & 0.82 & 4.17 & 76.88 & 18.25 & 0.70 \\
4 & 5.68 & 63.39 & 25.97 & 4.97 & 4.11 & 52.18 & 39.93 & 3.78 \\
5 & 2.15 & 67.07 & 24.50 & 6.28 & 1.57 & 55.65 & 37.97 & 4.81 \\
6 & 2.38 & 86.23 & 9.94 & 1.45 & 1.93 & 79.68 & 17.16 & 1.24 \\
7 & 3.12 & 81.63 & 11.63 & 3.62 & 2.51 & 74.59 & 19.85 & 3.05 \\
8 & 1.73 & 59.49 & 28.76 & 10.01 & 1.23 & 47.98 & 43.33 & 7.46 \\
9 & 3.71 & 86.75 & 7.30 & 2.23 & 3.08 & 82.07 & 12.90 & 1.95 \\
10 & 2.61 & 54.05 & 26.14 & 17.19 & 1.89 & 44.65 & 40.34 & 13.12 \\
11 & 3.21 & 63.75 & 25.93 & 7.10 & 2.32 & 52.44 & 39.85 & 5.39 \\
\hline
\end{tabular}
\caption{EDS results of the points in figure 1.}
\end{table}
composition basically consists of the same phases. Combined with the XRD data of sintered compacts (in figure 2), it can be found that the deep black areas of sintered compacts are $\alpha$-Fe solid solution, the light black areas are $\gamma$(Fe,Ni) solid solution, the dark gray areas are Cu$_{13.7}$Sn solid solution and the light gray areas are Cu$_3$Sn intermetallic compounds.

As shown in figures 1(c)–(e), the microstructures of Fe-based sintered compacts consist of four phases which are $\alpha$-Fe solid solution, $\gamma$(Fe,Ni) solid solution, Cu$_{13.7}$Sn solid solution and Cu$_3$Sn intermetallic compound when Sn content is 5 wt% or 10 wt%. There is no Cu$_3$Sn intermetallic compound in the microstructure when Sn content is 15 wt%. By comparison of the EDS results in figures 1(a)–(c), it is found that Ni content in Cu$_3$Sn phases and Cu$_{13.7}$Sn solid solution increase with the increase of Ni content in metal binders. That is because Cu and Ni exhibit the same crystal structure and similar lattice parameter, and both of them can form infinite solid solution[21]. More Cu atoms are replaced by Ni in Cu$_3$Sn phases and Cu$_{13.7}$Sn solid solution with the increase of Ni content. In addition, Ni and Fe can form $\gamma$(Fe,Ni) solid solution[22]. Powder sintering is different from conventional liquid phase solidification. The cooling process of powder sintering is non-equilibrium solidification, in consequence, there are still mass $\gamma$(Fe,Ni) solid solutions in the microstructure after cooling. The microstructure contains the mixture of $\alpha$-Fe and $\gamma$(Fe,Ni) phases when Ni content is higher, while the mixed structures are more uniform and finer.

### 3.2. Comparative analysis of the mechanical properties of Fe-based sintered compacts

The main function of metal binder is to reserve the diamond particles within the cutting layer of diamond tools. The strength of metal binder has a great influence on diamond retention. Moreover, the hardness of binder should match the wear rate of the workpieces to make good use of diamond particles. Hence the mechanical properties of metal binder control the performance of the tools in significant measure. The sintered compacts of metal binder must have equal strength and hardness.

The mechanical properties of Fe-based sintered compacts were determined. Table 3 lists the bending strength and hardness of sintered compacts. Through comparison of sample C, sample D and sample E, it can be

![Figure 2. XRD patterns of sample D of sintered compact.](image)

| Number | Hardness (HRB) | Bending strength (MPa) |
|--------|----------------|------------------------|
| A      | 98.94          | 797.03                 |
| B      | 100.29         | 755.78                 |
| C      | 99.72          | 827.02                 |
| D      | 95.12          | 1138.58                |
| E      | 99.67          | 400.07                 |
found that the hardness of sample D is 95.12 HRB, while the hardness increases as the Sn content increases. At the same time, the bending strength reduces significantly with the increase of Sn content. By comparing sample A, sample B and sample C, it can be noticed that the hardness of the sintered compacts increases with the rising Ni content, while the bending strength increases firstly and then decreases with the increase of Ni content.

Because of the low melting temperature of Sn metal, it formed liquid phase in the sintering process. The liquid phase can fill the interspace between metal powder. Besides, viscous flow and capillary force of liquid phase promote the slip and rearrangement of solid powder particles. Thus the density of sintered compacts is improved. Liquid phase of Sn interacts with Cu powder to form brittle Cu3Sn phases. The higher Sn content results in more brittle phases, so the hardness of sintered compacts improves with the increase of Sn content. However, more brittle phases can split the matrix, resulting in the rapid decrease of bending strength.

According to the EDS analysis of sintered compacts, Ni element can dissolve into Cu3Sn and Cu13.7Sn phases and that leads to solution strengthening. Ni element and Fe element can form γ(Fe,Ni) solid solution. Powder sintering is different from conventional liquid solidification. The cooling of powder sintering is a process of non-equilibrium solidification. Large amounts of γ(Fe,Ni) solid solutions exist in the microstructure of sintered compacts. As shown in figure 1, when the component of metal binder has lower Sn content and higher Ni content, there are a mass of mixed structures of α-Fe and γ(Fe,Ni) solid solutions and the morphology of the mixture is more uniform and fine. Therefore, proper increase of Ni content is beneficial to improve the bending strength of sintered compacts.

3.3. Crack initiation and propagation of sintered compacts
The microstructures of sintered compacts which have different components almost all contain α-Fe solid solution, γ(Fe,Ni) solid solution, Cu13.7Sn solid solution and Cu3Sn phases, while only the ratio of each phase in the microstructures is different. For this reason, partial sintered compacts were studied on crack initiation and propagation in matrix. Figure 3 shows the morphology of the cracks in sample C, sample D and sample E after bending the sintered compacts. As shown in figure 1(a), it can be known that the crack initiates from Cu3Sn phase and propagates in the Cu3Sn phase. The propagation of tiny crack is stopped by the Cu13.7Sn solid solution or Fe-based solid solution. As shown in figure 1(b), it is observed that the crack propagates along the interface between Cu3Sn phases and γ(Fe,Ni) solid solution. As shown in figure 1(c), it can be noticed that a large crack mainly propagates in the Cu3Sn phase and many small cracks develop around the large crack.

It follows from the above that the cracks initiate from brittle Cu3Sn intermetallic compounds and quickly propagate in the brittle phases. However, the propagation of crack can be impeded or stopped by Cu13.7Sn solid solutions which has good toughness. In addition, the cracks propagate easily along the boundary between Cu3Sn
intermetallic compounds and \(\gamma\)(Fe,Ni) solid solutions. Crystal structure of \(\text{Cu}_2\text{Sn}\) phases is orthorhombic with lattice constants \(a = 5.529\,\text{Å}, b = 4.323\,\text{Å}, c = 4.775\,\text{Å}\). The \(\gamma\)(Fe,Ni) phases have a face-centered cubic crystal structure with lattice constants \(a = 3.585\,\text{Å}\). There are great differences of the crystal structure and lattice constants of the two phases. They have large lattice misfit, only incoherent interfaces can be formed. That results in a weak interfacial combination between the two phases. The stress at the tip of the crack exceeds the interface bonding strength of two phases, resulting in the separation of interface and the initiation of induced crack. The induced crack and original crack propagate at the same time and connect, so the interfaces of \(\text{Cu}_2\text{Sn}\) phases and \(\gamma\)(Fe,Ni) phases split up.

To sum up, the hardness and bending strength of Fe-based sintered binders is decided by combined action of elements. The properties of metal binder must match the properties of the machined work piece, and metal binder matrix must have enough hold-force to diamond particles and certain wear to ensure the outcrop of cutting tool. The induced crack and original crack propagate at the same time and connect, so the interfaces of \(\text{Cu}_2\text{Sn}\) phases and \(\gamma\)(Fe,Ni) phases split up.

4. Conclusion

(1) The microstructure of Fe-based sintered compacts consists of \(\alpha\)-Fe solid solution, \(\gamma\)(Fe,Ni) solid solution, \(\text{Cu}_{13.7}\text{Sn}\) solid solution and \(\text{Cu}_3\text{Sn}\) intermetallic phases. However, there is no \(\text{Cu}_{13.7}\text{Sn}\) solid solution in the microstructures when Sn content is excessive.

(2) With the increase of Sn content, the hardness of the sintered compacts increases, while the bending strength reduces significantly. With the increase of Ni content, the hardness increases, while the bending strength increases firstly and then decreases.

(3) The cracks initiate from brittle \(\text{Cu}_3\text{Sn}\) intermetallic phases and quickly propagate in the brittle phases or along the interface between \(\text{Cu}_3\text{Sn}\) intermetallic compounds and \(\gamma\)(Fe,Ni) phases. \(\text{Cu}_{13.7}\text{Sn}\) solid solutions can impede the propagation of crack.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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