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# Fabrication of a diamond impregnated tool using iron nanopowder binder

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To replace the high cost material of cobalt matrix, the feasibility of diamond impregnated tools using structure-modified Fe nanopowder was investigated in terms of its compaction and related sintering properties. The structure was controlled that the spherical agglomerate shape for the easily rearrangement at low pressure range and the bimodal typed porous agglomerate structure are principally responsible for the green density improvement. The structure modified Fe nanopowder showed higher compactability than the structure non-treated Fe nanopowder during the compaction process and homogeneously dispersed diamond in the segment. After sintering, the structure modified Fe nanopowder showed sound segment shape and reached 93.7% T.D. without substantial grain growth. The characteristics of compaction and the sintered segments were evaluated in terms of the microstructure. Moreover, a prototype product was made and core-drilling test was carried out using the structure modified Fe nanopowder. The results are discussed in comparison with the standard of commercial products.

Key words: Iron nanopowder, Fe/Diamond composite, Compaction, Sintering, Microstructure.

### Introduction

Diamond impregnated tools composed of diamond particles embedded into a metal matrix is widely employed in grinding and cutting tools for the processing of hard materials such as ceramic, concrete and natural stone [1, 2]. The powder metallurgy (PM) process is the most promising route for fabricating a metal bonded diamond segment [3]. The fabrication process of diamond segments consists in mixing a metal matrix powder with diamond, cold pressing and finally sintering [2, 4]. During this process, the diamond particles are bonded with a metal matrix by means of a combination of chemical and physical interactions and also, it is necessary to control the sintering temperature below about 900 °C in order to avoid diamond graphitization due to its negative affect on the final cutting performance of the tool [4]. Moreover, the metal matrix must be tailored to wear at the same rate as the diamond, so that, when a diamond is worn during tooling and cutting, a new set is exposed to uniform cutting [5]. Thus, the metal matrix powder is required to have adequate wear resistance, good retention with diamond particles, and, most of all properties that enable low sintering [6].

Cobalt and cobalt-based alloy powders satisfy these requirements so they are used mostly for the production of the diamond segment as the matrix material [2, 7].

However, cobalt is subject to price fluctuations in industry, is a strategic material, and does not seem to be the best choice for application [6, 7]. For this reason, recent research has focused on Fe, Cu, and Ti powders to replace cobalt powder [8]. Among these elements, Fe is the most attractive material due to its suitable price and mechanical properties [7]. However, Fe powder still has several barriers to replace the cobalt matrix such as poor compactability and a high sintering temperature over 1100 °C.

Recently, the authors' group has developed spherical shaped iron nanopowder agglomerates with improved flowability and compactability by spray drying of ballmilled Fe<sub>2</sub>O<sub>3</sub> nanopowder and a subsequent hydrogen reduction process [9, 10]. This new type of Fe nanopowder achieved remarkable compaction density of over 70% theoretical density (T.D.) at conventional compacting pressure as well as full densification over 99% T.D. at low sintering temperature of 700 °C. Based upon this result, it is expected that Fe nanopowder as a binder material for diamond impregnated tools will provide a stable size tolerance of the segment and strong bonding of the composites during the consolidation process.

The present study has therefore attempted to investigate the feasibility of Fe nanopowder agglomerates as a binder material of diamond impregnated tool for replacing high price cobalt binder material. For this purpose, the entire fabrication process of the diamond-Fe nanopowder composites including compaction and consolidation by sintering was investigated. The results are discussed in terms of microstructure and mechanical property in comparison with commercial tool materials.

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# **Experimental Procedure**

Commercially available iron oxide powder (Fe<sub>2</sub>O<sub>3</sub>, 99.99%) and diamond powder (300-600 µm) were used as raw materials for Fe/diamond composite. The iron oxide powder was ball-milled in methyl alcohol at a speed of 2400 rpm for 10 hr. The slurry of the milledoxide powder was spray-dried by an argon-gas blast atomizer. The fine agglomerates of the Fe<sub>2</sub>O<sub>3</sub> nanopowder collected by the cyclone, and the coarse granules collected in the main chamber were discarded. The obtained Fe<sub>2</sub>O<sub>3</sub> agglomerates were reduced in hydrogen atmosphere at 450 °C and 550 °C for 1 h in order to have different microstructures of particle and pore distribution in the agglomerates. To prevent reoxidation during handling in air, wet-coating of the organic binder without milling media was performed on as-spray dried and reduced Fe nanopowder.

For comparison, the conventional Fe nanopowder was prepared by the same methods of previous study [9, 11]. Briefly, the slurry of milled oxide in 2400 rpm for 10 h was dried in oven at 50 °C for 10 h, and then sieved down to a 100 mesh size. This oven-dried powder was then reduced under a hydrogen atmosphere at 450 °C for 1 h. To control the agglomerate size and to prevent re-oxidation, a wet-milling technique using an organic binder was conducted on the as-reduced Fe nanopowder in ethyl alcohol. The compactability of these powders was evaluated by a compaction experiment using a cylindrical die under pressure ranging from 125 to 1250 MPa.

The synthesized Fe nanopowders were blended with diamond powder by a 3D-tubular mixer for 1 h. The content of the diamond powder was 7.5 vol.%, the identical content of conventional Fe/diamond composite for core-drilling. Then, the blended powder mixture was compacted into a segment shape for core-drilling by cold press at 350 MPa, which is a reasonable low pressure for an automated PM manufacture system [12]. The compacts were sintered at 800 °C for 2 hr in a hydrogen atmosphere. The sintering conditions were determined at a level to avoid diamond graphitization [13, 14] but also to be appropriate for mass production processes. The relative densities of the green- and sintered Fe/ diamond composites in the segment shape were measured by the Archimedes' method using distilled water. The microstructures of the powder and segment specimens were observed by optical microscopy and scanning electron microscopy (SEM). Also, the hardness of the sintered segments was characterized by the Rockwell B scale. The cutting performance of the fabricated Fe nanopowder segments was evaluated by fabricating a prototype core drill and a core-drilling test.

# **Results and Discussion**

Fig. 1 shows the SEM micrographs of Fe nanopowders

prepared in all conditions. The as-oven dried and 450 °C reduced powder shown in Fig. 1(a) is the same as that used in our previous study [9, 11]. The powder has an irregular agglomerate structure consisting of Fe particles of about 20-100 nm in size. On the other hand, the as-spray dried and 450 °C and 550 °C reduced powders (S1 and S2) in Fig. 1(a, b) have a uniform and spherical agglomerate structure with a size ranging from 1 to 5 µm. The Fe particles in the agglomerates of the S1 and S2 powders grew up during heat-treatment of hydrogen reduction to form bimodal size distributions; the particle size of S1 was 10-50 nm and 150-300 nm, and that of S2 was 10-50 nm and 250-500 nm, as shown in Fig. 1 (b) and (c). The distinguishing characteristics of these powders are summarized in Table 1. It is also observed that the S1 and S2 powders contain a large number of micro pores in the agglomerate. In general, the agglomerate powders fabricated by the spray drying method have approximately 40-60% lower density than the theoretical density of the material due to the intraagglomerate pores [15, 16]. This could contribute to the decreasing density difference between the Fe nanopowder (7.87 g/cm<sup>3</sup>) and the diamond powder (3.52 g/cm<sup>3</sup>), thereby decreasing the segregation problem due to the density difference in the powders during mixing [17]. In addition, the most important benefit of the spherical agglomerate powder lies in its increasing effect on the flow property during compaction by the low frictional structure of agglomerate.

In order to investigate the compaction properties of the Fe nanopowders, the relative density of the compacts made from the Fe nanopowders was measured under a pressure range of 65-1250 MPa, and the result is represented in Fig. 2. The relative densities of the compacts in all conditions increased with increasing applied pressure, until finally saturated over 1000 MPa. Under the entire pressure range the lowest green density near 50%T.D. appears in the WD powder whereas the S2 powder showed the best compactability over 80%T.D. at 1000 MPa. Such a discrepancy of the green densities can be interpreted in terms of the microstructural features of the powders. Firstly, the spherical shape of the nanopowder agglomerate enhances the sliding and rearrangement of the agglomerates during the initial stage of compaction. As a result, the relative densities of the S1 and S2 powders below 100 MPa are higher than that of the WD powder, indicating ease of rearrangement of the powders at low pressure range. Secondly, the bimodal typed porous agglomerate structure is principally responsible for the green density improvement. As discussed in Fig. 1 and Table 1, the agglomerate powders of S1 and S2 consist of fine and coarse nanoparticles with bimodal size distributions, where the coarse particles and fine particles are a majority in the S1 and S2 powder, respectively. Such bimodal size distributions can improve the packing density by substitution of large dense particles for porous clusters of small particles, and by the filling of Fabrication of a diamond impregnated tool using iron nanopowder binder



Fig. 1. SEM micrograph of Fe nanopowders: (a) as-oven dried and 450 °C reduced (WD), (b) as-spray dried and 450 °C reduced (S1) and (c) as-spray dried and 550 °C reduced (S2).

Sample	WD	S1	S2			
Drying method	Oven dried	Spray-dried	Spray-dried			
Reduction temperature (°C)	450	450	550			
Agglomerate structure	Irregular	Spherical	Spherical			
Agglomerate size (µm)	0.5-5	1-5	1-5			
Particle size (nm)	20-100	10-50 & 150-300	10-50 & 250-500			

Table 1. Characteristics of the starting powders

small particles into the interstices between the large particles [18]. As a result, the green parts of the S1 and S2 powders can reach higher density than 50%T.D. of the WD green part consisting of the monomodal particles. In addition, the saturated density values of S1 (61.5%T.D.) and S2 (81.2%T.D.) are in good agreement with emprical and simulation studies of random close packing of bimodal powder mixture [19, 20].

Based on the results of the compaction behavior, the Fe/diamond composites in the segment shape were fabricated using three starting Fe nanopowders with diamond powder as represented in Fig. 3. Also, the



Fig. 2. The relative density of compacts made from the Fe nanopowders: (a) as-oven dried and 450 °C reduced (WD), (b) as-spray dried and 450 °C reduced (S1) and (c) as-spray dried and 550 °C reduced (S2).

relative densities of the compacted and sintered segments with the amount of volume shrinkage are listed in Table 2. The compacted parts made from WD, S1 and S2 in Fig. 3(a-c) appear to be sound and the relative densities of the compacted segments were determined to



Fig. 3. Photographs of compacted and sintered segments of (a, d) as-oven dried and 450 °C reduced (WD), (b. e) as-spray dried and 450 °C reduced (S1) and (c, f) as-spray dried and 550 °C reduced (S2) Fe nanopowders.

**Table 2.** Compacted- and sintered density and volume shrinkageFe/diamond composite segment.

Sample	WD	S1	S2
Green density at 350 MPa (%T.D.)	38.6	44.1	63.2
Sintered density at 800 °C for 2 h (%T.D.)	-	83.4	93.7
Volume shrinkage (%)	_	39.2	30.5

be 38.6, 44.1, and 63.2%T.D., respectively, which is in fair agreement with the compaction results of Fig. 2. In the compacted part of WD in Fig. 3(a), however, quite a number of diamond particles are concentrated in a corner of the segment. This phenomenon is mostly attributed to different flowability between the WD powder and diamond powder. An extremely large surface area of Fe nanopowder is unfavorable to freeflowing during handling, leading to a delay of powder feeding in the mold compared to the diamond powder. Such an undesirable concentration of diamond particles may bring about not only an inhomogeneous density distribution causing defects in the segment during sintering but also degradation of performance as a diamond tool. In contrast, the compacted segments of S1 and S2 show a sound surface structure without concentration of diamond particles as seen in Fig. 3(b) and (c). This remarkable improvement in the distribution of the diamond particles in the matrix is undoubtedly owing to the structural modification of the Fe nanopowder agglomerates. In other words, the spherical shaped agglomerate increases the flowability by a low



**Fig. 4.** SEM micrographs of sintered segments using (a, b) asspray dried and 450 °C reduced (S1) and (c, d) as-spray dried and 550 °C reduced (S2) Fe nanopowders.

frictional structure and decrease the density mismatch between the Fe nanopowders and the diamond powder by porous structure [12].

After sintering, it was found that the WD segment has numerous large cracks through the entire body as can be seen in Fig. 3(d), so that the sintered density and volume shrinkage cannot be measured. In contrast to this, the sintered segments of S1 and S2 show a clear surface without any cracks or defects (Fig. 3(e, f)), and the sintered densities of both the segments reached 83.4 and 93.7%T.D., respectively. The results reveal that the sintered segment starting from higher green density achieves the higher sintered density with less volume shrinkage and structure stability. For the Fe/diamond sintered composite, it is well known that the thermal expansion mismatch between the diamonds and metal matrix induces residual stress at the interface [21, 22]. Moreover, it should be intensified when shrinking Fe matrix contains nonshrinking of diamond particles but rather thermally expending during pressureless sintering. In order to confirm this, the surface structures of the S1 and S2 sintered segments were observed by scanning electron microscopy (SEM).

It can be seen from the micrographs of Fig. 4(a) and (b) that the microcracks in the segments are generated at the boundary between the diamond particles and the Fe matrix, and propagated along the edge of diamond particles. Furthermore, the surface of the diamond particle is exposed, indicating that wetting and adhesion of the diamond to the Fe matrix is poor [23]. These results imply that the residual stress between the diamond particles and elastic Fe matrix, produced during sintering, is higher than the fracture stress of the matrix. However, it should be noted here that the crack length of the S2 segment is much shorter than that of the S1 segment of about 200 µm and the diamond particles are mostly covered with the matrix. That is, even though crack generation is inevitable, it can be substantially suppressed by increasing the green density. Obviously, the homogeneous density distribution and the close powder packing in the compacted segments are responsible for decreasing the shrinkage and the residual stress despite the pressureless sintering [24].

Fig. 4(b) and (d) shows the matrix structures of the S1 and S2 segments. It can be seen that the grain size of the S1 and S2 segments were about 400 nm and 500 nm, respectively, which is not so different from the initial particle size. Also, fine pores are found mostly at the grain boundaries among the grains. Considering the sintered densities in Table 2, the sintering process of S1 segment (83.4%T.D.) is on the intermediate stage whereas that of the S2 segment (93.7%T.D.) on the final stage of sintering [12]. Such rapid densification is well explained in terms of nanopowder sintering. As reported in previous investigations [9], the densification kinetics in the intermediate sintering stage are governed by the material transport process along grain boundaries at low temperature where the contribution of the volume diffusion to the densification might be ignored compared to that of the grain boundary diffusion. In this study, as discussed in Fig. 1, the S1 and S2 powders consist of nanoparticles characterized by a bimodal size distribution. Therefore, these nanoparticles could effectively increase the number of grain boundaries with a hierarchical interface structure consisting of fine and coarse boundaries that act as the preferred diffusion paths for densification [25, 26].

For the Fe/diamond composite tool, the hardness of the metal matrix is one of the most important properties concerned. If the hardness of the matrix is relatively low,



Fig. 5. Comparison of the Rockwell B hardness of the segments.

 Table 3. Perforating test results of the prototype core drill made from the S2 segment.

Sample	Drilling speed (cutting depth per time, cm/min)	Tool life (Cutting depth per wearing length cm/mm)
S2 segments	4.59 (79.6%)	140.65 (64.2%)

the matrix is unable to retention the diamond particles and they are pulled out before it sufficiently acts as its cutting role [27, 28]. In contrast, when the metal matrix becomes so hard, it cannot expose the cutting edges of the diamond particles easily during cutting [23]. In order to examine the hardness of the fabricated segments, the Rockwell B scale hardness was measured and represented in Fig 5. While the S1 segment has an HRB of  $48.9 \pm 8.5$ , the S2 segment showed an HRB of  $99.0 \pm 2.4$  due to the higher density. Comparing the hardness values with those of the cobalt matrix used for the diamond impregnated tool, the S1 segment is insufficient as a tool due to the lower hardness originating from the microcracks and its low density. In the case of the S2 segment, the hardness has a similar value as that of the conventional cobalt matrix. This result indicates that the Fe/diamond sintered segment has enough hardness to be applied in core-drilling tooling. In order to confirm this, the prototype core drill was fabricated using S2 segments. Nine pieces of produced S2 segments were welded to the steel core with an outer diameter of 100 mm and a thickness of 3.5 mm. With this prototype, perforating tests were performed on the reinforced concretes.

Table 3 shows the results of the perforating test using the prototype core-drill made from the S2 segments. For the comparison, the percentages of the performance for the commercial cobalt product are also indicated in parentheses. It can be seen that the fabricated segment showed a slightly lower drilling speed than the cobalt segment, indicating that the diamond particles were easily pulled out from the Fe matrix despite the similar hardness. This may arise from the poor adhesion property of the Fe matrix on diamond causing microcracks near the diamond particles as seen in Fig. 4(c). The tool life time of the S2 segment is also lower than that of cobalt, as can be expected from the sintered density. However, there are possible solutions to solve these problems. Firstly, a depositing strong carbide-forming metal on a diamond surface can dramatically promote adhesion between the diamond particles and the Fe matrix. According to the literature [23, 29-31], the carbide layer behaves as a diffusing barrier to avoid excessive reaction of the matrix with the diamond particles, and a buffer zone to reduce thermal stress between them. Presently, standard grades of titanium-coated diamond grits are commercially available, which can improve the mechanical properties of metalbonded diamond tools. Secondly, control of the sintering condition or pressure assisted sintering can improve the mechanical properties by decreasing the shrinkage and effective densification process. Thus, it can be concluded that the use of structure modified Fe agglomerate nanopowder in this study is an effective way to replace cobalt based diamond composite.

#### Conclusions

The fabrication of diamond impregnated tools using structure-modified Fe nanopowder was investigated in terms of the compaction and related sintering properties. It was found that the segment fabricated by the as-oven dried Fe nanopowder (WD) had severe diamond segregation during the compaction process due to poor compactability. Consequently, such an undesirable concentration of diamond particles brought about not only an inhomogeneous density distribution causing defects in the segment during sintering but also degradation of the performance as a diamond tool. Consequently, WD was not suitable to be applied to the diamond segment. In contrast, the as-spray dried and 450 °C and 550 °C reduced Fe nanopowders (S1 and S2) showed higher compactability than that of WD powder. It is indicated that the spherical agglomerate shape for easily rearrangement at low pressure range and the bimodal typed porous agglomerate structure is principally responsible for the green density improvement. For this reason, the segment showed sound body with a homogeneously dispersed diamond. After sintering, the sintered segments of S1 and S2 showed a clear surface without any cracks. The sintered densities of both segments reached 83.4 and 93.7%T.D., respectively. In the microstructure analysis, the grain size of the S1 and S2 segments was about 400 and 500 nm, respectively, which is almost the same as the initial particle size. However, the crack length of the S2 segment is much shorter than that of the S1 segment of about 200 µm and the diamond particles are mostly covered with the matrix. It can be substantially suppressed by increasing green density because of decreasing shrinkage value with increasing dimensional reliability. A prototype product was made and a core-drilling test was carried out by using the S2 segments. The results of the S2 segments showed only 80% for cutting depth per time and 64.2% cutting depth per wearing length than the commercial product. However, the results are significant because these values can be improved by including alloying elements or control of compaction and sintering conditions to improve the mechanical property by decreasing the shrinkage and effective densification process. Thus, we conclude that the use of structure modified Fe nanopowder is an effective alternative to replace cobalt based diamond composite.

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