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Influence of pore size on toughness of Fe-Mo-Cu-C sintered and carburized components

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Abstract
The influence of pore size on the toughness of Fe-Mo-Cu-C sintered and carburized components was investigated. The charpy impact value of the component made from Fe-0.4%Mo diffusion-alloyed steel powder mixed with 2%Cu powder and 0.3% graphite powder significantly increased with a decrease in pore size. The highest impact value of 17 J/cm^2 was obtained at the average pore size of 12 µm. This value was 25% higher than that of the sintered and carburized component made from conventional Fe- 4%Ni-1.5%Cu-0.5%Mo alloyed steel powder called 4Ni even though the component was made from a Ni-free alloyed steel powder. The mechanism of the improvement of the impact value by pore size refinement and the influence of microstructural difference between the components made from Fe- 0.4%Mo diffusion-alloyed steel powder and 4Ni on toughness is discussed based on optical microstructural observation and crystal orientation analysis data.
Keywords:

Sintering, Carburized

1. Introduction

Sintered steel components are used in numerous applications, such as the automotive field, because of their complex shapes, high dimensional accuracy, economically and easy producing near-net shape form. Recently, alloyed steel powders for the sintered compacts with higher strength and toughness is required in terms of lighter weight and more compact size in parts. Previously, sintered and carburized components (carburized components) made from 4% Ni alloyed steel powder (4Ni), in which 4% Ni, 1.5% Cu and 0.5% Mo are diffusion-alloyed the surface of iron powder, were used for the application. However, because of supply instability and high price of Ni, a Ni-free alloyed steel powder for high strength and high toughness carburized components has been required.

Against this background, Unami et al. [1)] reported that carburized components having strength equal to that of 4Ni can be obtained by using Fe-Mo-Cu mixed powder, in which Cu powder is added to Fe- 0.45%Mo. However, the toughness of the carburized components made from the powder was one-half that of carburized components made from 4Ni.

Densification is an effective approach for improving the toughness of sintered components [2)]. Although this improvement is thought to occur because densification of the compact reduces the number of pores that become points of origin for fracture and also reduces the size of pores, the individual contributions of porosity reduction and pore size refinement have not been clarified. Therefore, the influence of pore size refinement on toughness was investigated by producing carburized components with the same density and different pore sizes using Fe-Mo-Cu mixed powder.

2. Experimental Procedure

2.1 Fabrication

The raw material powder properties, chemical compositions, and detail of additives are shown in Table 1. Raw material powders A to E are Fe-0.4%Mo diffusion-alloyed steel powders, and raw material powders A to D were prepared by sieving raw material powder E in order to controlling pore size of sintered specimen. Raw material powder F is a 4Ni (Fe-4.0%Ni-1.5%Cu-0.5% Mo diffusion-bonded alloyed steel powder). Raw material powders A to E were mixed with 2.0% electrolytic copper powder, 0.3% natural graphite powder and 0.5% of a high density lubricant [3)], then sample powder A to E was obtained. Raw material powder F was mixed with 0.3% graphite powder and 0.8% zinc stearate, then sample powder F was obtained.

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These sample powders were compacted into a bar shape (bar specimen) with dimensions of 10 mm (width) x 10 mm (height) x 55 mm (length). The compacting pressure of each sample powder was adjusted to obtain the bar specimens with the sintered density in the range of 7.05 ± 0.05 Mg/m3. These bar specimens were sintered at 1130°C for 20 min under an endothermic gas atmosphere by a mesh belt furnace. Using some of the sintered bar specimens, smooth round bar specimens with a parallel portion diameter of 5 mm and length of 15 mm were prepared by machining. These sintered bar and round bar specimens were carburized at 870°C for 60 min in an atmosphere with a carbon potential of 0.8%, quenched in oil at 60°C and tempered at 180°C for 60 min, then carburized bar and round bar specimens were obtained. The sintered specimens and carburized specimens were designated sintered specimens A to F and carburized specimens A to F from the symbols of the raw material powders used.

2.2 Evaluation

The density of the sintered bar specimen was measured by the Archimedes method based on JIS Z 2501. A tensile test was conducted based on JIS Z 2241 using the carburized round bar specimen. A notchless charpy impact test was performed based on JIS Z 2242 using the carburized bar specimen. In addition, the fracture surfaces of the carburized bar specimens after the impact test were observed by scanning electron microscopy (SEM).

The pore distribution of cross section perpendicular to the longitudinal of the carburized bar specimens were observed by optical microscopy. The area *A*, peripheral length *P* and equivalent circle diameter *d* of each pore in the micrographs was measured by using the image analysis program Image J. The accumulated area fraction, in which the area of the pores are accumulated from the small *d* side, is also calculated as a function of *d*, and the point where this accumulated area fraction reaches 50% of the total area fraction of pores is defined as the average pore diameter d_{50}. Circularity of pore was also calculated from the measured pore area *A* and peripheral length *P* with equation (1).

$$C = \frac{4\pi A}{P^2} \quad \cdot \cdot \cdot (1)$$

Circularity is an index of pore shape, and shows the maximum value of 1 in the case of a perfect circle. After the observation of the pore distribution, the specimens were etched with nital, microstructure of the specimen was also observed by optical microscopy.

After the microstructural observation by optical microscopy, a crystal orientation analysis was carried out by the electron backscatter diffraction (EBSD) method using a field emission scanning electron microscope (FE-SEM). The analyzing area and the step of EBSD measurement were 115 µm x 80 µm and 0.2 µm, respectively. The distribution of the bcc and fcc phases was measured from the obtained data. In addition, Ni concentration mapping of the carburized spesimen F was conducted with an electron probe microanalyzer (EPMA).

Table 1 sample powder properties and additives

Sample ID	Raw powder properties		Diffused alloy element / mass%			Addtives / mass%		
	particle size distribution / µm	Average particle diameter D_{50} / µm	Mo	Cu	Ni	Cu powder	Graphite powder	Lubricant
A	-180/+106	143.0	0.4	-	-	2.0	0.3	0.5 (High density)
B	-106/+75	90.5						
C	-75/+45	60.0						
D	-45	22.5						
E	-180	66.3						
F	-180	86.3	0.5	1.5	4.0	-		0.8 (Znst)

3. Results

The optical micrographs of the carburized components with as-polished were shown in Fig. 1. The pore size of carburized specimen D is smaller than that of carburized specimen A. The relationship between average particle diameter of raw material powder D_{50} and average pore diameter of carburized specimen d_{50} was shown in Fig. 2. The D_{50} and d_{50} of carburized specimen A to E decreased with a decrease in D_{50}. Therefore, the pore size was controlled by controlling the particle size distribution.

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The relationship between d_{50} and charpy imact vale is shown in Fig. 3. The charpy impact value increased with a decrease in d_{50}. And it showed that the impact value increases with a decrease in the average pore diameter, even when the sintered densities of the specimens were same. Comparing carburized specimens B and F, which had approximately the same average pore diameter, the charpy impact value of carburized specimen F (4Ni) was higher than that of carburized specimen B.

The relationship between d_{50}, the tensile strength and the breaking elongation in the tensile test was shown in Fig. 4 and 5. The tensile strength and the breaking elongation increased with a decrease in d_{50}. Comparing carburized specimens B and F, which had approximately the same average pore diameter, the tensile strength of carburized specimen B was higher than that of carburized specimen F (4Ni), but the breaking elongation of the B was lower than that of the F.
Optical micrographs of the carburized specimens with nital etching were shown in Fig. 6. In carburized specimen E, the microstructure consisted mainly of martensite with a trace amount of the ferrite phase. On the other hand, in the carburized specimen F (4Ni), the microstructure consisted of two phases, namely, martensite and a white-colored region which is Ni-enriched region.

The micrographs of SEM observation of the fracture surfaces of the carburized specimens after impact value test is shown in Fig. 7. Although all the specimens fractured at the sintering neck, and the fracture surface displayed a dimple pattern.

4. Discussion

The fracture surfaces of all the carburized specimens after the impact test displayed a dimple pattern, as shown in Fig. 7. It indicates that ductile fracture occurred at the sintering neck part in all cases[4]. The relationship between breaking elongation in the tensile test and the charpy impact value is shown in Fig.
8. These plots have linear relathioship, and the impact values increase with an increase in breaking elongation. Therefore, in this research, the toughness is affected the ductility of microstructure. Hence, the influence of pore size and microstructure on ductility is discussed respectively.

Pores in sintered steel are a point of origin for fracture[2] because stress concentrates around pores when sintered materials are deformed. In the case of elliptical pores, as shown in Fig. 9, this kind of stress concentration is expressed by the following equation[5].

$$\sigma_k = K\sigma \cdot\cdot\cdot (2)$$

where σ is uniform stress applied perpendicular to the major axis of an ellipse, σ_k is stress generated at the tip of the major axis side (point A in the figure) and K is a stress concentration factor. The stress concentration factor K is expressed as follows by using the minor axis a and the major axis b of an elliptical pore.

$$K = \frac{2b}{a} + 1 \cdot\cdot\cdot (3)$$

In eq. (3), b/a is replaced as the aspect ratio R, then eq. (4) is obtained.

$$K = 2R + 1 \cdot\cdot\cdot (4)$$

From eq. (4), K increases in flatter ellipses with larger values of R, that is, K increases in a decrease in circularity of the ellipse. The stress concentration generated by a pore changes depending on R, which is an index of the pore shape, independent of the pore size. Therefore, the relationship between the pore diameter and pore circularity, which is an index of pore shape, is shown in Fig. 10 for carburized specimen A, which had the largest average pore diameter, and carburized specimen D, which had the smallest. In both carburized specimens, pore circularity increases with a decrease in pore diameter. Furthermore, in carburized specimen D, the number of pores with coarse diameters and low circularity was smaller in comparison with A. Aspect ratio R in the above-mentioned elliptical pore and circularity

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C is not equal. However, since pores with low circularity *C* are assumed to have complex shapes, and these pores has high curvature like the edge of the major axis of a flat ellipse with high aspect ratio *R*. Therefore, the pores with lower circularity have higher stress concentration factor *K*. Accordingly, in comparison with carburized specimen A and D, carburized specimen D contains fewer pores with a large stress concentration factor *K* compared with these of carburized specimen A. The ductility of carburized specimen D improved because the decrease in the number of pores of this type also reduces the number of points of origin for fracture.

On the other hand, in spite of the fact that the pore sizes of carburized specimen B and F were same, carburized specimen F displayed larger elongation. Hence, the microstructures of these two carburized specimen were analyzed in detail by a crystal orientation analysis using the SEM/EBSD method. Figure 11 shows the phase distribution maps of carburized specimen B and F. Almost the entire region of carburized specimen B consists of a bcc phase, on the other hands, a fine fcc phase exists in the vicinity of the pores in carburized specimen F. The mapping of the distribution of Ni by EPMA is also shown in Fig. 11. The Ni-enriched regions coincide with the regions where the fine fcc phase exists. From these facts, the fcc phase is a retained austenite phase[6] that was formed as a result of Ni concentration.

Retained austenite and martensite are microstructures with different hardness and ductility. Comparing the two,martensite is a high hardness phase with poor ductility[7], whereas retained austenite has the effect of imparting ductility to the microstructure as a result of plastic deformation and the accompanying strain-induced transformation, as is known to occur in the TRIP (transformation induced plasticity) phenomenon[8] in steel sheets. In carburized specimen F, retained austenite formed accompanying Ni concentration in the vicinity of pores. As mentioned above, the area around pores is considered to a point of origin for fracture by stress concentration. Thus, if a soft phase such as a retained austenite phase forms in the vicinity of a pore, plastic deformation occurs preferentially accompanying stress concentration, and as a result, the stress concentration in the pore area is relieved. This is the reason that the charpy impact value of carburized specimen F is higher than that of carburized specimen B, which did not contain a soft phase, even though the pore diameters of the two materials were same.

5. Conclusion

The pore size on toughness of Fe-Mo-Cu-C sintered and carburized components was investigated. The conclusion is as follow;

(1) Fe-Mo-Cu-C sintered and carburized specimen with different pore sizes were prepared by controlling the particle size of the raw material powder. In these specimens, the charpy impact value, tensile strength and total elongation increased with a decrease in pore size.

(2) The charpy impact value of the Fe-Mo-Cu-C sintered and carburized specimen with the pore diameter of 13.5 μm showed toughness equal to that of the conventional sintered and carburized specimen made from the Fe-Ni-Mo-Cu-C (4Ni).

(3) The fracture surfaces of all the sintered and carburized specimen after the impact test were the ductile fracture surface type with a dimple pattern. There was also a high correlation between the charpy impact value and the breaking elongation in the tensile test.

(4) In the Fe-Mo-Cu-C sintered and carburized specimens, the number of irregularly-shaped coarse pores with low circularity decreased with a decrease in pore size.

(5) From (3) and (4), the increase in the charpy impact value by a decrease in pore size of the Fe-Mo- Cu-C sintered and carburized components is the result of a decrease in stress concentrations in the vicinity of pores due to the decrease in the number of coarse, irregularly-shaped pores, and the accompanying improvement in ductility.

(6) Comparing the charpy impact values of Fe-Ni-Mo-Cu-C sintered and carburized components and the Fe-Mo-Cu-C sintered and carburized components with same pore diameters, the Fe-Ni-Mo-Cu-C sintered and carburized components displayed higher impact values.

(7) It is considered that a retained austenite phase forms accompanying Ni concentration in the vicinity of pores in Fe-Ni-Mo-Cu-C sintered and carburized components, and as a result, stress concentrations are relieved, ductility is improved, and the charpy impact value increases.

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