Effect of Ceramic Dispersions on Sintering and Mechanical Properties of P/M Steel-Ceramic Composites

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Sintered low alloy steel components are used for various commercial applications. Inferior strength is a limitation of sintered low alloy steel powder metallurgy products in many applications. In order to improve mechanical properties including toughness, steel-ceramic composites are being developed. In the present investigation, 2% Cu steel and its composites with ceramic particles or fiber are studied. Premixed steel powders with 4 vol% of $\text{Al}_2\text{O}_3/\text{SiC}$ particles or Al_2O_3 fiber were sintered at 1150°C under ENDO (Endothermic) gas atmosphere for 1 h. Sintered properties like densification parameter, sintered density, porosity etc. have been measured. Mechanical properties evaluation including hardness, tensile strength have also been conducted. Fractography was studied under SEM. Results of the experiments are discussed in details.

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I. Introduction

Components made from sintered low alloy steel are generally used in automotive industry, domestic appliances, electric motors, printers etc. because of the economy. Low alloy steel containing 2% Cu is very popular grade steel which is used for automobile parts. Strength and toughness of the material are two important requirements for automotive components in addition to homogeneity of structure and narrow tolerance limit.

It is important to enhance the strength and toughness of the material. Many oxides like Al_2O_3 , ZrO_2 , MgO , Y_2O_3 , TiC etc. have been used by many research workers⁽¹⁾⁻⁽⁹⁾. Alumina is very hard and stable oxide, so it is selected to one of the dispersoids to increase the strength. Fiber or particulate was selected to find their effect on ductility of the alloy. SiC is also selected to compare their role with Al_2O_3 before developing the commercial alloy. Earlier work⁽¹⁰⁾ with stainless steel based matrix showed that optimum properties were obtained with 4 vol% dispersoid. Therefore, in the present investigation 4 vol% dispersoid is selected for each composition. The aim of the present investigation is to increase the strength of the steel matrix by introducing a second phase as a form of particles or fibers.

II. Experimental Procedure

Raw materials used in the present study are given below.

Iron: Water atomized iron powder WPL-200 (Hoganas). Characteristics of the materials as follows:

Chemical composition	Mass percentage
Carbon	0.02
Silicon	0.05
Manganese	0.2
Sulphur	0.015
Phosphorous	0.02
Iron	Balance
Hydrogen loss	0.2%
Average particle size	63 μm
Apparent density	2.6 Mg/m^3
Flow rate	35 s/50 gm
Compressibility	6.95 Mg/m^3 at 600 MPa

Al_2O_3 fiber: The characteristics and the composition of the fiber (dupont) as follows:

Composition	99% Al_2O_3
Tensile modulus	378 GPa
Tensile strength	1378 MPa
Melting point	2318 K
Filament diameter	20 μm
Cross section	Round
Length/mass	Approx. 4702 km/m^3
Density	3.9 Mg/m^3

Al_2O_3 particle: The composition and particle size are as follows:

Al_2O_3 particle	
Composition	99.4% pure
Ave. particle size	1 μm

SiC particle: The composition and particle size are listed below.

SiC particle	Composition
SiC	98.5%
SiO ₂	0.5%
Si	0.3%
Ave. particle size	0.5 μm

Copper powder: The composition of copper powder is given below.

Copper powder	99.6% pure
Ave. particle size	44 μm

Carbon powder: The chemical purity and particle size are as follows:

Carbon powder (graphite-grade)	99.9% pure
Ave. particle size	1 μm

The premixed steel powder was prepared by mixing iron, copper and carbon powder in a mixer for 1 h. The mixer was special type having vertical rotational axis. The final steel powder has the composition of low alloy steel, which is as follows:

Copper : 2%
Carbon : 0.8%
Iron : Balance

Mixing of premixed steel powder and 4 vol% of ceramic particles or discontinuous fibers were performed in the same mixer for 1 h in each composition. Al₂O₃ fibers chopped to the length of approximately 4–6 mm prior to mixing, so that the brittle fiber does not break into small pieces if the length was very large. Length was decided to keep 4–6 mm length to get desired fiber aspect ratio. Dog-bone type green compacts were compacted in hydraulic press using a compaction pressure of 450 MPa. Figure 1 shows the dimension of the Dog-bone specimen.

Sintering was performed in a mesh-belt type industrial furnace at a temperature of 1423 K for 1 h under ENDO gas atmosphere. The composition and dew point of the endothermic gas (ENDO) is as follows:

H₂: 38%
CO: 16.3%
N₂: 45.1%
Methane: 0.6% Dew point 261 K.

Fig. 1 Dimension of Dog-bone specimen.

Densities of the compacts were measured using water displacement method as well as from physical dimension. Sintered porosities were calculated from theoretical density and actual sintered density of the alloys. Theoretical density was calculated using the following formula:

$$100/D_{\text{mix.}} = (\text{mass\% A})/D_A + (\text{mass\% B})/D_B$$

$D_{\text{mix.}}$ = Density of composite

D_A = Density of component A

D_B = Density of component B.

Densification parameter was determined using the formula given below.

$$\Delta D = \frac{\text{Sintered density} - \text{Green density}}{\text{Theoretical density} - \text{Green density}}$$

Hardness of sintered samples were measured in Rockwell hardness testing machine using B-scale. Tensile test of the sintered compacts were carried out in 1195-Instron machine, using a chart speed of 10^{-2} m/min. Cross-head speed of the machine was 10^{-3} m/min. Percentage elongation was calculated from final and initial gauge length (25 mm) values.

Metallography studies were carried out under Scanning Electron microscopes. Fractography of the selected samples were observed under SEM. For X-ray mapping, X-ray was collected for 5 h and then distribution of particles in the matrix was highlighted with the help of software.

III. Experimental Results

1. Sintered density

The steel compact without any second phase shows the maximum sintered density amongst all the materials (Fig. 2).

Sintered porosity of different compositions is plotted

Fig. 2 Sintered density of steel and its composites.

Fig. 3 Sintered porosity of steel and its composites.

Fig. 4 Densification parameter of sintered composites.

in Fig. 3. It is maximum for 4 vol%Al₂O₃ fiber containing composite.

2. Densification parameter

Densification parameter of steel and its composites are depicted in Fig. 4. Densification parameter is maximum for SiC containing composite. Steel without any dispersoid shows lower value than any of its composites. Densification parameter for fiber containing composite is lower than particulate composites.

3. Hardness

Hardness values of composites (Fig. 5) are higher than that of straight steel compact. It is generally more for Al₂O₃ and SiC particle containing composite. Hardness value is also higher for fiber containing composite than the steel compact, although the sintered porosity is maximum for the fiber containing composite.

Fig. 5 Hardness of sintered steel and its composites.

Fig. 6 Tensile strength of sintered steel and its composites.

Table 1 Percentage elongation of sintered steel and its composites.

Material	Percentage elongation
Sintered steel	3.5
Steel + Al ₂ O ₃ particle	0
Steel + SiC particle	0
Steel + Al ₂ O ₃ fiber	3

4. Tensile strength

The tensile strength behaviour (Fig. 6) of the composites is similar to hardness. Tensile strength improvement is quite substantial for 4 vol%Al₂O₃ containing composite followed by 4 vol%Al₂O₃ fiber. In general, strength of steel is improved by the presence of Al₂O₃ or SiC particles or fiber in the present experiments. From the tensile

Steel + Al_2O_3 particle

Steel

Fig. 7 Distribution of Al_2O_3 particles in steel matrix after mixing.

Steel + SiC particle

Fig. 8 Distribution of SiC particles in steel after mixing.

Steel + Al_2O_3 fiber

Fig. 9 SEM micrograph of sintered samples.

test experiments, it is found (Table 1) that percentage elongation decreases in presence of second phase dispersed in the composites except of 4 vol% Al_2O_3 fiber containing one which shows 3% elongation as compared to original 3.5% of straight sintered steel.

5. Microstructure

Figures 7 and 8 show the distribution of Al_2O_3 and SiC particles respectively in the steel matrix. Bright particles are Al_2O_3 and SiC respectively. These figures indicate the fair distribution of ceramic particles in the steel matrix. Figure 9 shows the SEM micrographs of sintered Steel-Ceramic composites. The micrograph of the steel is typical 0.8% carbon steel. The bright particles unfortunately came from the polishing abrasive particles. Suspended alumina was used as polishing media. It is observed from the SEM micrograph, that the distribution of Al_2O_3 fiber for 4 vol% composite is fairly uniform. Figure 10 shows the X-ray mapping of the Steel-SiC composites. It is observed that the distribution of SiC particles (black globule in the grey matrix) in the matrix is well distributed.

X-ray mapping of steel + SiC

Fig. 10 X-ray mapping of sintered steel-SiC composite.

6. Fractography

Fractographs of different samples are shown in Fig. 11. It is noticed that all the particulate composites have cleavage type of fracture. Steel compact without any second phase shows some dimple in the fractograph. In the case of fiber containing composite fracture is less cleavage in nature than other particle containing composites.

Fig. 11 Fractographs of sintered steel and its composites. (a) Steel, (b) Steel at higher magnification, (c) Steel+ Al_2O_3 particle, (d) Steel+SiC particle, (e) Steel+ Al_2O_3 fiber.

IV. Discussion

1. Sintering behaviour

It is evidenced from the present study that densification parameter are higher for composites as compared to straight steel. This behaviour indicates that there is some chemical reaction taking place in between matrix and dispersoid in the present system. This type of activated sintering was reported⁽¹¹⁾⁻⁽¹³⁾ in the case of stainless steel-ceramic particulate composites system. Reaction of SiC with iron-based alloys are also reported elsewhere⁽¹⁴⁾.

2. Mechanical properties

It is observed that hardness values are higher for the composites. This is due to dispersion hardening effect of

the composites which is further confirmed from lower hardness value of the fiber containing composite where no dispersion hardening is expected.

The positive effect of ceramic second phase dispersion in the steel matrix is clearly observed from the improvement of tensile strength of the composites. It is maximum for fine Al_2O_3 particle containing composite and followed by 4 vol% Al_2O_3 fiber containing one. Higher strength of alumina containing composite as compared to SiC containing composite may be due to the fact that fine size SiC particles may agglomerate to some extent during mixing and thus may affect the properties to some extent. From the results, it appears that Al_2O_3 particle containing composite is optimum for the present system to have the maximum benefit of dispersion strengthening effect. Al_2O_3 fiber is able to carry load from the matrix

and hence improve the strength. This implies that the increase in composite strength is the result of particulate/fiber strengthening effect. In addition, grain sizes of the composites are more or less smaller than matrix and it also contribute to increase the strength to some extent. Percentage elongation up to 3% for 4 vol% Al_2O_3 fiber containing composite as compared to no elongation for particulate containing composites indicates that toughness property of the material will definitely be improved with fiber addition. This is further evidenced from the fractographs where fiber containing composite shows less cleavage fracture than other compositions.

V. Conclusions

The following conclusions can be drawn from the present study.

- (1) Densification parameter of composites is higher than steel.
- (2) Porosity is maximum for fiber containing steel.
- (3) In general, hardness of the composites are higher for particulate composites as compared to steel or fiber containing composite.
- (4) Tensile strength of the material increases in presence of ceramic phase. It is maximum for Al_2O_3 particle containing composite.
- (5) Ductility decreases to zero in presence of second phase particles. However, 3% elongation is achieved in the case of Al_2O_3 fiber containing composite.

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