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## Research Report

# Development of Cu- and Ni-free Sintered Steels with Excellent Strength and Toughness through Microstructural Control

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**BABSTRACTII** In this study, Cu- and Ni-free high-strength sintered steels with excellent impact energy were developed using Si and Mn (FeMS powder [Fe-69 wt%Mn-20 wt%Si-1.1 wt%C]) and Cr (SUS410L powder [Fe-13 wt%Cr]) for strengthening. The microstructure-property correlation in these steels revealed that the volume fraction of martensite ( $V_f^M$ ) had to be maintained between 15 and 35% to obtain an excellent combination of tensile strength and impact energy: over 1000 MPa and around 50 J/cm<sup>2</sup>, respectively. The  $V_f^M$  increased gradually with increasing amount of SUS410L powder. This suggests that the addition of SUS410L is sufficient for precise microstructural control of Cu- and Ni-free high-strength sintered steels. Good combinations of strength, hardness and impact energy were obtained for fine and homogenous distributions of island-shaped martensite through the addition of SUS410L, which appears to contribute to the high impact energy.

**KEYWORDSII** Cu- and Ni-free Sintered Steels, High Strength and Toughness, Microstructural Control

# 1. Introduction

Cu- and Ni-containing high-strength sintered steels with tensile strengths of over 1000 MPa have been widely used for heavy-duty automotive parts. However, there has been a strong demand for the development of Cu- and Ni-free high-strength sintered steels from the viewpoint of cost-effectiveness and recyclability.<sup>(1-4)</sup> For example, Fe-Mn-Mo-C and Fe-Cr-Mo-V-C alloys have been developed as Cu- and Ni-free steels.<sup>(5,6)</sup> However, these showed limited impact energy, despite having strengths comparable to those of Cu- and Ni-bearing steels.

We have reported that Fe-Mn-Si-C alloys with a fine pearlitic structure<sup>(7,8)</sup> and tensile strengths of about 800 MPa showed higher impact energies than conventional Fe-Cu-C sintered steels. Our results suggested that Mn and Si addition in conjunction with microstructural refinement was a promising way to simultaneously obtain high strength and high impact energy without adding Cu and Ni.

This paper investigated Cu- and Ni-free sintered steels with excellent combinations of tensile strength (> 1000 MPa) and impact energy ( $\sim$ 50 J/cm<sup>2</sup>), focusing on microstructural control of the bainite-martensite

dual-phase structure to achieve the desired mechanical properties.

# 2. Experimental

The sample compositions studied are listed in **Table 1**. Fe-0.85 wt%Mo low-alloyed steel powder with a size of less than 150  $\mu$ m (**Fig. 1**(a)), FeMS powder

Sample	FeMS powder (wt%)	Graphite powder (wt%)	SUS410L powder (wt%)	Fe-0.85%Mo powder
1	0	0.6	0	Bal.
2	0.5			
3	1.0			
4	1.25			
5	1.5			
6	1.75			
7	2.0			
8	2.5			
9	3.0			
10	1.0		1.0	
11			2.0	
12			3.0	
13			4.0	
14			5.0	

 Table 1 The proportion of ingredients in the samples studied.

(Fe-69 wt%Mn-20 wt%Si-1.1 wt%C) with a size of 1.1  $\mu$ m in D<sub>50</sub> (Fig. 1(b)), natural graphite powder with a size of 5  $\mu$ m in D<sub>50</sub> and SUS410L powder (Fe-13 wt%Cr) with a mean particle size of 10  $\mu$ m (Fig. 1(b)) were used to obtain high-strength steels consisting of bainite-martensite dual-phase structures. The amount of FeMS powder was varied from 0 to 3.0 wt% to change the volume fraction of martensite. The amount of graphite powder was fixed to 0.6 wt%. The SUS410L powder was added, at 1.0 to 5.0 wt% to the samples containing 1.0 wt% FeMS and 0.6 wt% graphite.

Cylindrical specimens for density measurement (diameter: 23 mm; height: 10 mm), tensile specimens (**Fig. 2**), and Charpy impact specimens  $(10 \times 10 \times 55 \text{ mm})$ 



Fig. 1 SEM images of raw powders: (a) Fe-0.85%Mo low-alloyed steel powder, (b) FeMS powder, and (c) SUS410L powder.



Fig. 2 Dimensions of the plate specimen used for tensile testing.

were prepared by a warm compaction process at 423 K with die wall lubrication using water-dispersed lithium stearate<sup>(9)</sup> and sintering at 1423 K for 1.8 ks under a pure nitrogen atmosphere, followed by tempering at 473 K for 3.6 ks in air. After sintering, the average cooling rate from 1173 to 673 K was 0.8 K/s. The density of each cylindrical specimen was determined from its dimensions and weight. The tensile and impact properties and Vickers hardness were measured at ambient temperature. The microstructure was observed by optical and scanning electron microscopy. The volume fraction of martensite  $(V_f^M)$ was calculated using image analysis software. The distributions of alloying elements were measured by a field-emission-type electron probe micro-analyzer (FE-EPMA).

# 3. Results and Discussion

# 3.1 Variation in Mechanical Properties with FeMS Amount

**Figure 3** shows optical micrographs of sintered and tempered samples containing various amounts of FeMS powder from 0 to 2.0 wt%. The dark spots



**Fig. 3** Optical micrographs for sintered and tempered samples containing various amounts of FeMS powder: (a) 0 wt%, (b) 1.0 wt%, and (c) 2.0 wt%.

correspond to pores. Sample 1, which contained no FeMS powder, showed a fully bainitic structure. Sample 3, which contained 1.0 wt% FeMS, exhibited a bainite-martensite dual-phase structure with a  $V_{\rm f}^{\rm M}$  of 4%. The  $V_{\rm f}^{\rm M}$  of sample 7, which contained 2.0 wt% FeMS, was as high as 70%. **Figure 4** shows the change in  $V_{\rm f}^{\rm M}$  with FeMS amount. The rapid increase in  $V_{\rm f}^{\rm M}$  with increasing FeMS amount from 1.0 to 2.0 wt% is noteworthy.

The density measurement results for green and sintered samples are given in **Fig. 5**. The density decreased with increasing FeMS amount in both green and sintered samples. More porosity was observed in sample 7 than in samples 1 and 3 (Fig. 3).

**Figure 6** shows the variation in mechanical properties with FeMS amount. The tensile strength of the FeMS-free sample 1 was as low as 683 MPa.

The tensile strength increased with increasing FeMS amount up to 1.75 wt%, then decreased. The peak tensile strength was 1243 MPa for sample 6. Since the hardness increased monotonically with increasing FeMS amount, the observed decrease in tensile strength for > 1.75 wt% FeMS was presumably due to poor ductility. The impact energy improved slightly upon FeMS addition up to 0.5 wt%, decreasing with further increase in FeMS amount. The samples containing 1.25 to 1.5 wt% FeMS showed good combinations of tensile strength and impact energy, over 1000 MPa and about 48 J/cm<sup>2</sup>, respectively. These excellent mechanical properties appear to be attributable to the optimal volume fraction of martensite and relatively low porosity.



**Fig. 4** Variation in the volume fraction of martensite with the amount of FeMS.



Fig. 5 Change in density with the amount of FeMS.



**Fig. 6** Variation in mechanical properties with FeMS amount.

# 3. 2 Microstructural Control by Addition of SUS410L Powder

**Figure 7** shows the variations in mechanical properties with the amount of SUS410L powder. All samples contained 1.0 wt% FeMS powder and 0.6 wt% graphite powder. The strength and hardness gradually increased with increasing SUS410L amount, maintaining excellent impact energies of around 50 J/cm<sup>2</sup>. **Figure 8** shows the relationship between the SUS410L amount and  $V_{\rm f}^{\rm M}$ . The increase in  $V_{\rm f}^{\rm M}$  with the amount of SUS410L is more gradual than the increase in  $V_{\rm f}^{\rm M}$  with the amount of FeMS (Fig. 4). A scanning electron micrograph and a Cr distribution map obtained from the same region of sample 11, which contains 2.0 wt% SUS410L, are presented in



Fig. 7 Variation in mechanical properties with SUS410L amount.

**Fig. 9**. The  $V_{\rm f}^{\rm M}$  was 20%. It is interesting to note that the Cr-enriched regions correspond to island-shaped martensite. This suggests that the volume fraction of fine island-shaped martensite can be controlled by varying the amount of SUS410L powder. The Cr-enriched regions are thought to be formed mainly at the triple junctions of coarse low-alloyed steel powders, as illustrated in **Fig. 10**.



Fig. 8 Variation in volume fraction of martensite with SUS410L amount.



Fig. 9 (a) Optical micrographs and (b) Cr distribution map taken from the same region in sample 11.



Fig. 10 Schematic of microstructural control through powder preparation techniques.

## 3.3 Microstructure-property Correlation

**Figure 11** plots the mechanical properties as a function of  $V_{\rm f}^{\rm M}$  for all the samples. The tensile strength and hardness were found to be governed by  $V_{\rm f}^{\rm M}$ . To obtain a tensile strength higher than 1000 MPa,  $V_{\rm f}^{\rm M}$  had to be greater than 15%. The impact energy also had a strong correlation with  $V_{\rm f}^{\rm M}$ , which had to be less than about 35% to get impact energies around 50 J/cm<sup>2</sup>. Therefore, in order to obtain an excellent combination of tensile strength and impact energy, i.e., over 1000 MPa and around 50 J/cm<sup>2</sup>, respectively,  $V_{\rm f}^{\rm M}$  had to be maintained in the range from 15 to 35%. For  $V_{\rm f}^{\rm M}$  below 50%, samples containing SUS410L tended to show slightly higher impact energies than samples without SUS410L at the same  $V_{\rm f}^{\rm M}$ . Fine and homogenous distributions of island-shaped martensite, formed by the addition of SUS410L (Fig. 10), appear to contribute to the high impact energy.

As shown in Fig. 4,  $V_{\rm f}^{\rm M}$  increased rapidly from 4 to 70% as the amount of FeMS was increased from 1.0 to 2.0%. By contrast, as the amount of SUS410L was increased from 0 to 5%,  $V_{\rm f}^{\rm M}$  increased only gradually (Fig. 8). This indicates that precise microstructural control, in particular, control of  $V_{\rm f}^{\rm M}$  in the range from 15 to 35%, is achieved more easily by controlling the amount of SUS410L, rather than FeMS.

Figure 12 shows the relationships between tensile strength, hardness and impact energy. The square dots represent the typical performance of conventional high-strength sintered steel containing Cu and Ni. Good combinations of strength, hardness and impact energy



Fig. 11 Correlation between volume fraction of martensite and mechanical properties.



Fig. 12 Combinations of mechanical properties: (a) Tensile strength vs impact energy, and (b) hardness vs impact energy.

were obtained in the present SUS410L-containing steels. The steels developed in this study are thought to be promising candidates for replace conventional high-strength steels containing Cu and Ni.

# 4. Conclusions

Cu- and Ni-free high-strength sintered steels with excellent impact energy were developed by using Si and Mn (FeMS powder [Fe-69 wt%Mn-20 wt%Si-1.1 wt%C]) and Cr (SUS410L powder [Fe-13 wt%Cr]) for strengthening. The main conclusions were:

- (1) The microstructure-property correlation in Cuand Ni-free high-strength sintered steels revealed that the volume fraction of martensite ( $V_{\rm f}^{\rm M}$ ) had to be maintained between 15% and 35% to obtain excellent combinations of tensile strength (> 1000 MPa) and impact energy (~50 J/cm<sup>2</sup>).
- (2)  $V_{\rm f}^{\rm M}$  was very sensitive to the amount of FeMS powder.  $V_{\rm f}^{\rm M}$  increased from 4 to 70% as the amount of FeMS was increased from 1.0 to 2.0 wt%. Precise microstructural control through control of the amount of FeMS appeared to be difficult.
- (3) V<sup>M</sup><sub>f</sub> increased gradually with increasing amount of SUS410L powder. This suggests that SUS410L is sufficient to enable precise microstructural control of Cu- and Ni-free high-strength sintered steels.
- (4) Good combinations of strength, hardness and impact energy were obtained in the SUS410L-containing steels developed in the present study. The fine and homogenous distributions of island-shaped martensite formed by the addition of SUS410L appear to contribute to the high impact energy.

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### Figs. 2-3 and 5-6

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