

Improved Densification of Carbonyl Iron Compacts by the Addition of Fine Alumina Powders

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An investigation of the effect of alumina particles on the sintering behavior of a carbonyl iron powder compact was carried out in this study. Two different-sized alumina, 0.05 and 0.4 μm , were added to the iron compact at amounts up to 1.2 wt pct. When 0.4 μm alumina particles were added, no sintering enhancement was observed. But, in contrast to previous results reported in literature, the addition of 0.1 to 0.2 wt pct of 0.05 μm alumina particles was found to improve the densification. With 0.1 wt pct, the sintered density increased from 7.25 to 7.40 g/cm^3 after the compact was sintered at 1350 $^\circ\text{C}$ for 1 hour in hydrogen. Dilatometric curves showed that alumina impeded the early-stage sintering of iron in the α phase, but improved densification in the γ phase at high temperatures. These results, along with microstructural analysis, suggested that alumina particles exhibit dual roles; their physical presence blocks the diffusion of iron atoms, thus causing inhibition of sintering, while their grain-boundary pinning effect prevents exaggerated grain growth of iron and helps densification. It follows that, depending upon the amount and size of the alumina powders, either an increase or decrease in the final sintered density can be obtained.

I. INTRODUCTION

CARBONYL iron powders are frequently used in making powder injection-molded (PIM) compacts, which require high sintered densities. However, the density of the compacts for these applications is usually not fully dense after sintering.^[1,2,3] One of the main reasons for attaining such less-than-desired densification is that the exaggerated grain growth usually occurs when iron transforms from the α phase to the γ phase.^[4-7] The exaggerated grain growth causes pore isolation from grain boundaries. Once these pores are trapped inside the grain, they cannot be eliminated within a practical timeframe.

To improve the sintered density of carbonyl iron compacts, the main challenge is to inhibit the grain growth, particularly during the phase transformation. One of the common approaches is to add inert dispersoids into the iron in the hopes that they will impede the grain-boundary migration. However, most previous studies indicated that inert oxides caused an inhibition of sintering.^[8-11] Corti and Cotterill^[8] mixed γ alumina particles, which were smaller than 0.03 μm , with carbonyl iron powders by dry ball milling for 24 hours. The powder mixture was then pressed and sintered. The amount of alumina employed ranged from 0.16 to 16.0 wt pct, after which a marked inhibition of densification was observed. This study attributed the inhibition effect to the restricted diffusion of iron atoms due to the presence of alumina particles on the interparticle iron-iron contacts. Singh and Houseman^[9] and Singh^[10] studied the effect of alumina, titania, and zirconia on the densification of carbonyl iron powders between 1300 $^\circ\text{C}$ and 1490 $^\circ\text{C}$. The particle size of the three oxides varied from 0.005 to 0.040 μm , and the amount employed was between 0.5 and 2.0 wt pct. All three oxides inhibited densification. Studies on iron catalysts

also showed inhibited sintering by the addition of inert oxides.^[11] Other observations on the inhibited sintering of copper, nickel, and cobalt were also noticed, as summarized in a review article by Ashby *et al.*^[12]

Although most studies reported that inert oxides retarded densification of metal powders, Imai and Miyazaki^[13] demonstrated that the volume diffusion of silver increased slightly when it was mixed with 2 wt pct Al_2O_3 . Lu *et al.*^[14] reported an improved sintered density of PIM iron compacts when aluminum stearate was added in the binder system. The stearate was found to form Al_2O_3 during debinding and sintering and helped densification. Comparing these previous studies, the different results on the effects of alumina could be caused by the differences in the size and the amounts of alumina particles and/or the process (press-and-sinter molding or PIM). The purpose of this study was, thus to re-examine the effects of alumina on the sintering behavior of carbonyl iron powders. This study used only the press-and-sinter process and concentrated on the effects of the amount and size of alumina powders. The amount of alumina used ranged from 0.1 to 1.2 wt pct, and two sizes, 0.05 and 0.4 μm , were compared. Also, both wet and dry mixing techniques were employed to compare the effect of the uniformity of the alumina distribution. The results showed that an improvement in sintered density can be obtained by adding a small amount of 0.05 μm alumina particles.

II. EXPERIMENTAL PROCEDURE

Most carbonyl iron powders contain 0.5 to 1.0 wt pct carbon, which is a critical element in influencing the sintering behavior of iron. Thus, to avoid the complexity inherent in analyzing the sintering results of this study, the iron powder used was a reduced grade (CIP-R-1430, ISP Corp., Wayne, NJ) which contained only 0.065 wt pct carbon. It had an average particle size of 7 μm , and its characteristics are given in Table I.

Two types of alumina, 0.40 μm α -alumina and 0.05 μm γ -alumina, were used as the additive. Although the structures

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Table I. The Characteristics of Carbonyl Iron Powders Used in This Study

Powder designation	CIP-R-1430
Surface area, m ² /g	0.336
Pycnometer density, g/cm ³	7.84
<i>d</i> ₅₀ , μm	7
<i>d</i> ₉₀	15
<i>d</i> ₁₀	3.1
Carbon	0.065 pct
Nitrogen	0.006 pct
Oxygen	0.420 pct
Supplier	ISP Corp. (Wayne, NJ)

of these alumina particles are different, both of them are considered to be chemically inert to iron. Thus, the results and discussions presented in the following sections are related to their particle size alone. To attain a uniform oxide distribution in the iron matrix, alumina powders were dispersed in alcohol first and then mixed with iron powders to form a slurry, using a mortar and pestle. The slurry was dried, ground to -325 mesh powders, and then pressed into 55 pct dense pellets, 12 mm in diameter and 5 mm in thickness, using the floating die technique. No lubricant was used. The green compacts were heated at a rate of 10 °C/min and then isothermally sintered at 1200 °C and 1350 °C, respectively, for 1 hour in hydrogen.

To understand the effect of alumina on the sintering behavior of carbonyl iron powders, dilatometry analysis was employed to monitor the dimensional change of the compact during sintering. To examine the microstructural evolution, specimens were removed at different stages during heating. For optical microscopy analysis, sintered compacts were infiltrated with epoxy and cured prior to grinding and polishing, so that the pore shape would not be distorted. For scanning electron microscopy (SEM) examination, specimens were immersed in liquid nitrogen first and then fractured. With this method, little plastic deformation occurred, and the true internal structure of the compact, particularly the neck morphology, was retained.

III. RESULTS

A. Sintered Density

The sintered densities of specimens containing different amounts of 0.05 μm alumina particles are shown in Figure 1. At 1200 °C, the density of carbonyl iron compacts increased from 6.55 to 6.65 g/cm³ with a 0.1 wt pct alumina addition. As the temperature increased from 1200 °C to 1350 °C, the density also increased from 7.25 g/cm³ of the pure iron to 7.40 g/cm³ with 0.1 wt pct of alumina. In contrast, when 0.40 μm α-alumina was added, the density of the compact decreased at both 1200 °C and 1350 °C, as shown in Figure 2.

These results are different from those reported in References 9 and 10, which showed decreased densities in all tests. The difference could be caused by the alumina or the mixing methods employed. To simulate the dry mixing method used by Singh and Houseman,^[9,10] 0.05 μm alumina particles were mixed with carbonyl iron powders by dry ball milling for 24 hours. Figure 3 shows that the sintered densities thus obtained decreased as the alumina content

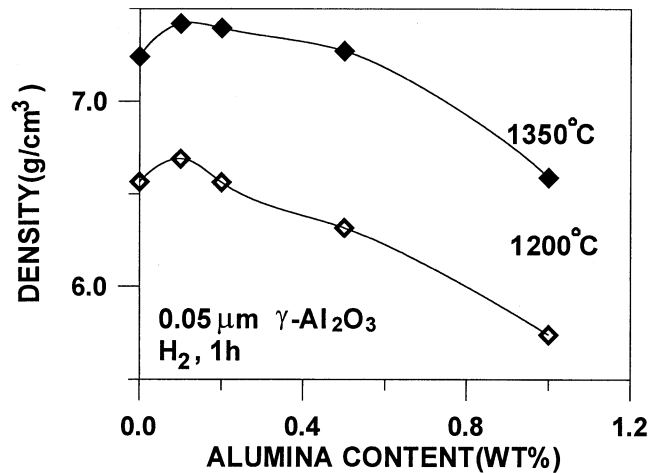


Fig. 1—The sintered densities of iron compacts containing different amounts of 0.05 μm alumina.

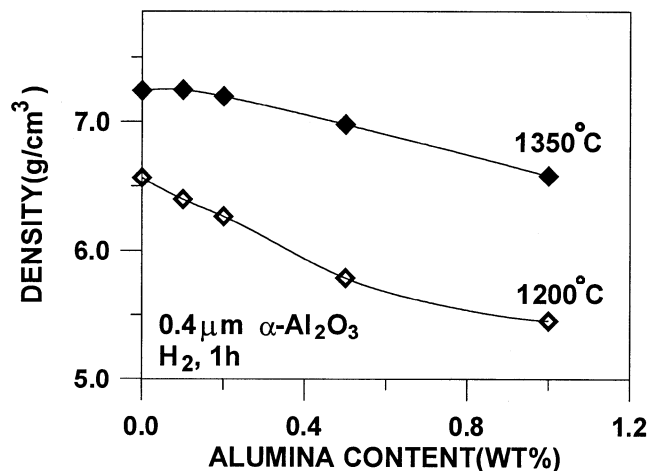


Fig. 2—The sintered densities of iron compacts containing different amounts of 0.40 μm alumina.

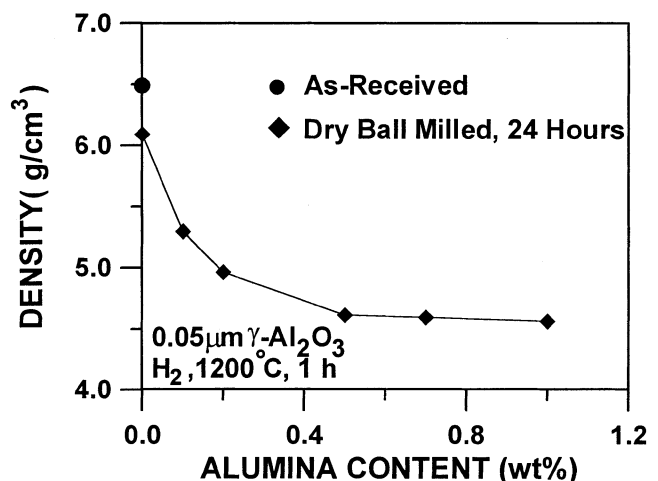


Fig. 3—The sintered densities of alumina-containing compacts prepared by the dry ball-milling method.

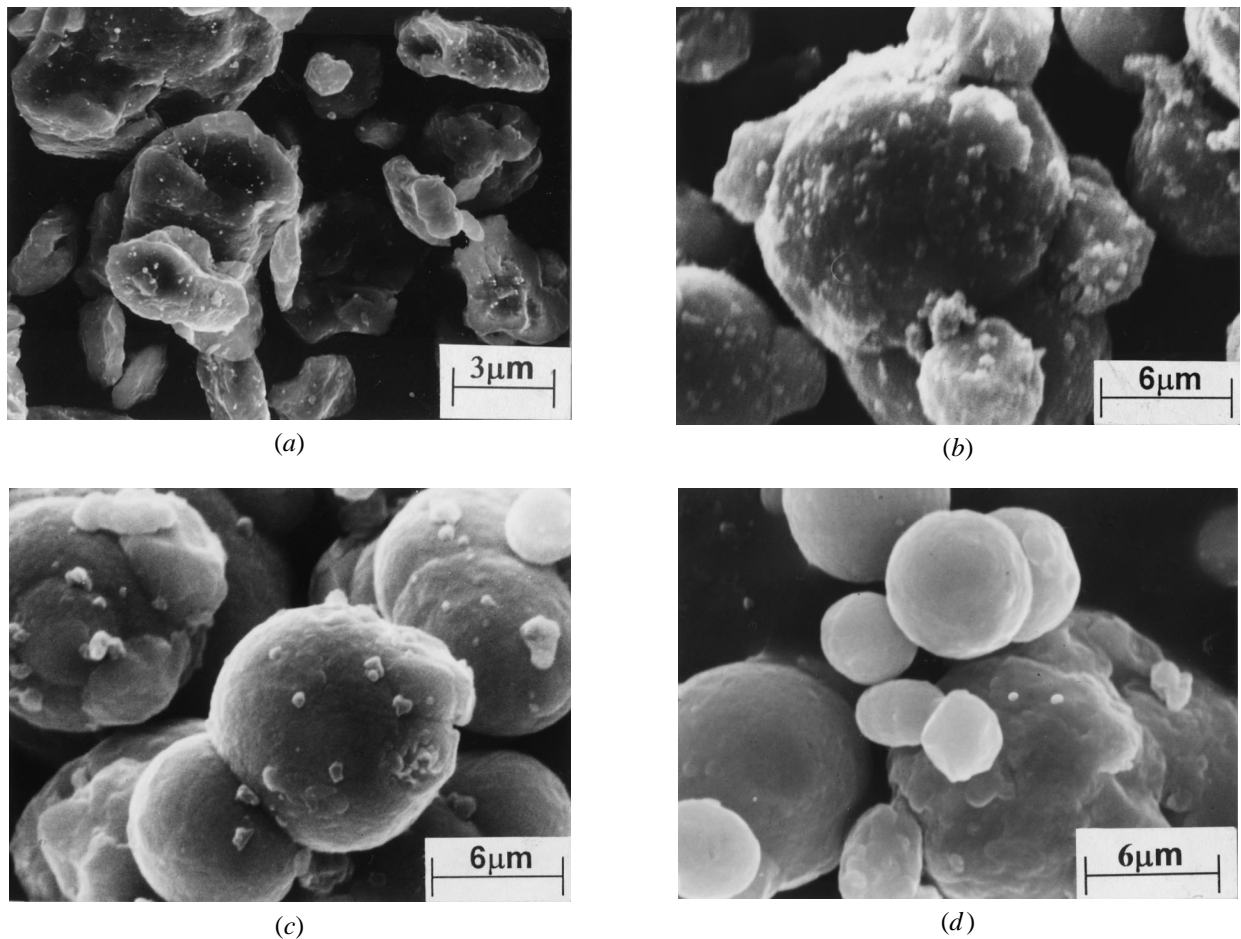


Fig. 4—The alumina particle distribution in (a) dry ball-milled powders containing 1 wt pct $0.05 \mu\text{m}$ alumina, (b) wet-mixed powders containing 1 wt pct $0.05 \mu\text{m}$ alumina, (c) wet-mixed powders containing 1 wt pct $0.4 \mu\text{m}$ alumina, and (d) as-received carbonyl iron powders.

increased, just as reported in Singh and Houseman's studies.^[9,10] The pure iron compact also shows a decrease in sintered density with dry ball-milled powders. Figure 4 compares the morphology of the mixed powders, which contained 1 wt pct $0.05 \mu\text{m}$ alumina particles. When the dry mixing technique was used, some alumina particles were embedded on the iron powder surface, and the iron particles became more irregular in shape, as shown in Figure 4(a). In contrast, the wet-mixed powder mixtures with 1 wt pct of $0.05 \mu\text{m}$ alumina and $0.4 \mu\text{m}$ alumina, as shown in Figures 4(b) and (c), respectively, illustrate that most alumina particles were uniformly distributed. The iron powder also retained its original size and shape, as shown in Figure 4(d).

B. Dilatometry Analysis

To better understand the effect of alumina particles on the sintering behavior of iron compacts, dilatometry tests were performed on specimens with and without alumina additions. Figure 5 shows that the density of the pure iron compact increased significantly with the increase in temperature in the α phase. But, the densification rate decreased dramatically after the phase transformation, due to the exaggerated grain growth. By adding 0.1 wt pct of $0.05 \mu\text{m}$ alumina particles, the sintering rate in the α phase was impeded. However, enhanced sintering was observed after

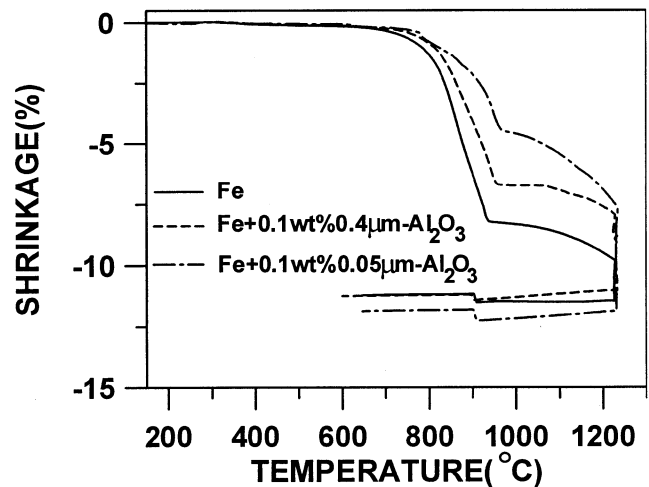


Fig. 5—Dilatometer curves of iron compacts with and without alumina additions.

the phase transformation, and the final density was greater than that without the alumina addition. Similar inhibition was observed during sintering in the α phase on the specimen containing $0.4 \mu\text{m}$ alumina. However, no sintering improvement was obtained in the γ phase.

Figure 6 compares the effect of the amount of alumina

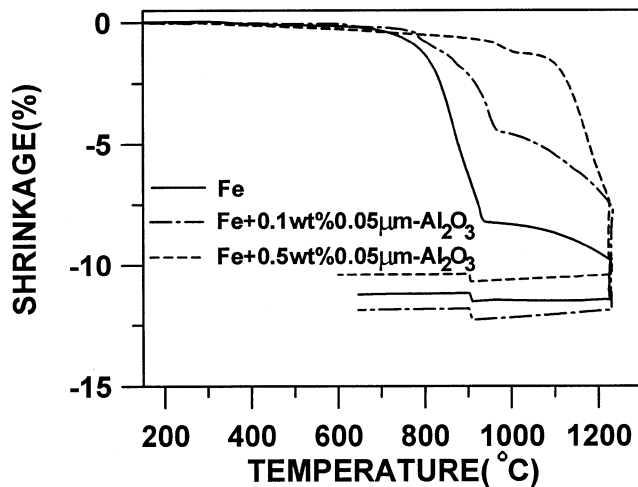


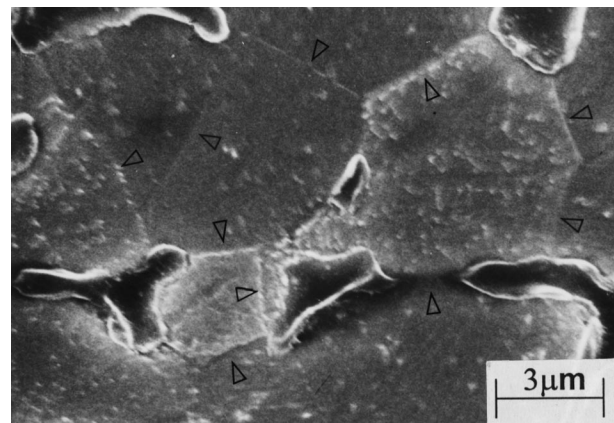
Fig. 6—The effect of the amount of 0.05 μm alumina on the sintering behavior of carbonyl iron compacts.

on the sintering behavior of iron compacts. As the amount of alumina increased from 0.1 to 0.5 wt pct, very little sintering was observed in the α phase, and the densification-rate change, which usually occurs at the phase transformation, became even unnoticeable. Significant densification did not occur until 1100 $^{\circ}\text{C}$. The final sintered density was lower than that of the pure iron compact.

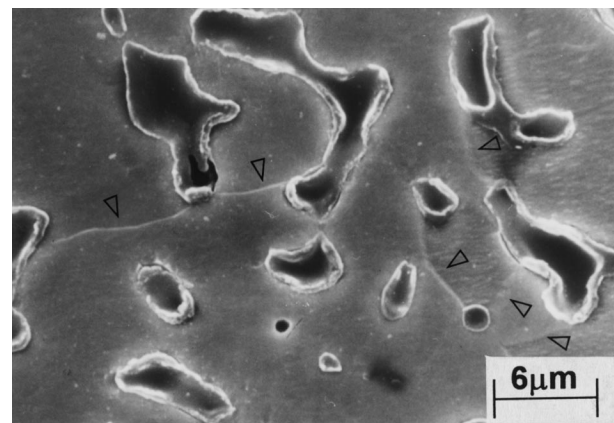
C. Microstructure

Since exaggerated grain growth is the most critical factor in influencing the final sintered density of carbonyl iron compacts, the evolution of the microstructure during heating was monitored. Figure 7(a) shows the microstructure of a pure iron specimen heated to 890 $^{\circ}\text{C}$, in which most pores are still connected to the grain boundaries. As the temperature increased to 950 $^{\circ}\text{C}$, significant grain growth occurred, leaving isolated pores inside the grain, as shown in Figure 7(b). Figure 8(a) demonstrated that, when 0.1 wt pct of 0.4 μm alumina was added, the compact that was heated to 950 $^{\circ}\text{C}$ showed slightly less grain growth than that of pure iron, shown in Figure 7(b), and most interparticle necks were still clearly discernible. But, exaggerated grain growth and trapped pores were still apparent. Thus, similar to iron, the densification rate was significantly impeded after the phase transformation, as shown in Figure 5. When 0.1 wt pct of 0.05 μm alumina was added, the inhibition effect on grain growth and neck growth became more significant, as shown in Figure 8(b). This helped improve the shrinkage rate in the γ phase, as shown in Figure 5.

The specimens that had been heated to 950 $^{\circ}\text{C}$ were also fractured after being immersed in liquid nitrogen. Figure 9(a) shows that the fractured areas in pure iron compacts were quite large, and the interparticle necks were no longer discernible. On the other hand, alumina-containing compacts tended to fracture at the necks, particularly in the compact that contained 0.1 wt pct of 0.05 μm alumina, as shown in Figure 9(b). This further confirmed that sintering was hindered by alumina particles and that smaller necks were obtained. Figure 9(c) shows that the fracture surface of a specimen containing 0.4 μm alumina had a mixture of both large transgranular and small neck areas. These evolutions



(a)



(b)

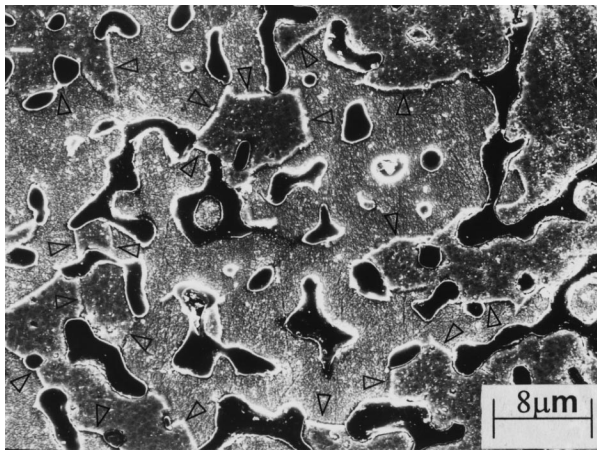
Fig. 7—The microstructure of pure iron compacts after being heated to (a) 890 $^{\circ}\text{C}$, showing pores attached to the grain boundary (indicated by arrows); and (b) 950 $^{\circ}\text{C}$, showing pores trapped inside the grains.

of the microstructure correspond to the sintering behavior of the compacts shown in Figure 5.

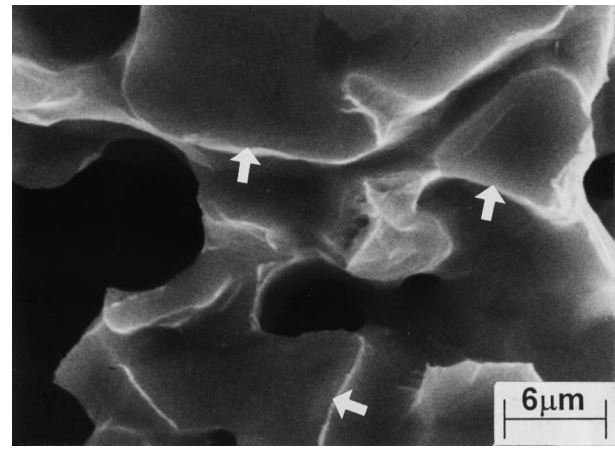
Figure 10(a) shows the microstructure of pure iron specimens sintered at 1350 $^{\circ}\text{C}$ for 1 hour in hydrogen. Considerable grain growth was observed, and the growth was in agreement with the results reported in previous literature.^[4–7] In contrast, compacts with either 0.4 or 0.05 μm alumina additions, as shown in Figures 10(b) and (c), respectively, reveal smaller grain sizes.

To observe the morphology change of the alumina powder in the iron matrix, 1 wt pct of 0.05 and 0.4 μm alumina, respectively, were mixed with iron powders. The mixed powders were poured into an alumina boat and were sintered at 1200 $^{\circ}\text{C}$ and 1350 $^{\circ}\text{C}$, respectively, for 1 hour in hydrogen. The lightly sintered compacts were fractured and then examined under SEM. Compared to the alumina particle sizes shown in Figures 4(b) and (c), Figure 11 illustrates that, after being heated to 1200 $^{\circ}\text{C}$ and 1350 $^{\circ}\text{C}$, the alumina particles coarsened, particularly the 0.05 μm alumina. This coarsening phenomena was also reported in previous literature.^[15,16,17]

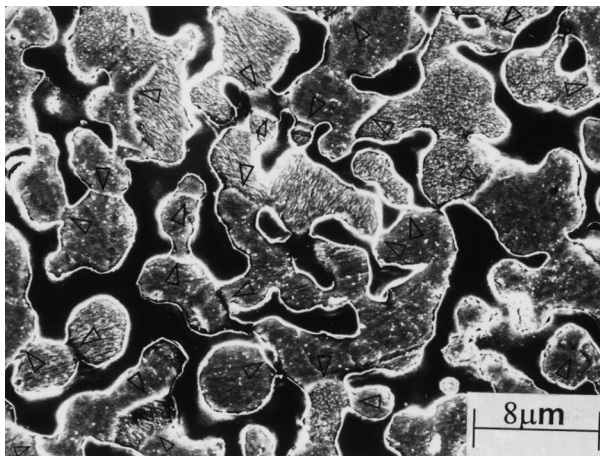
The effects of alumina particle size on the sintering behavior of carbonyl iron powder, as shown previously, can be summarized in Table III. With the same amount of alumina (0.1 wt pct), finer particles are more effective in inhibiting sintering in the α phase. At high temperatures in the γ phase,



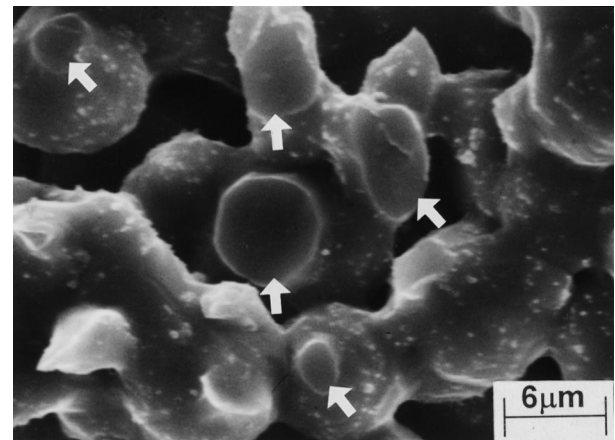
(a)



(a)



(b)



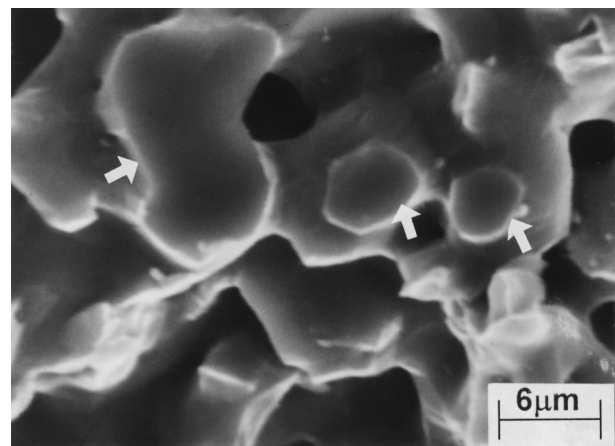
(b)

Fig. 8—The microstructure of alumina-containing iron compacts after being heated to 950 °C (a) with 0.1 wt pct 0.4 μm alumina and (b) with 0.1 wt pct 0.05 μm alumina.

there is less exaggerated grain growth, less pore isolation from grain boundaries, and more pronounced alumina coarsening, which are all beneficial for sintering. However, there is a limited range of the amount of alumina which aids in sintering, and only within that range can the final density be improved. When coarse alumina particles (0.4 μm) are employed, no improvement in sintered density can be found.

IV. DISCUSSION

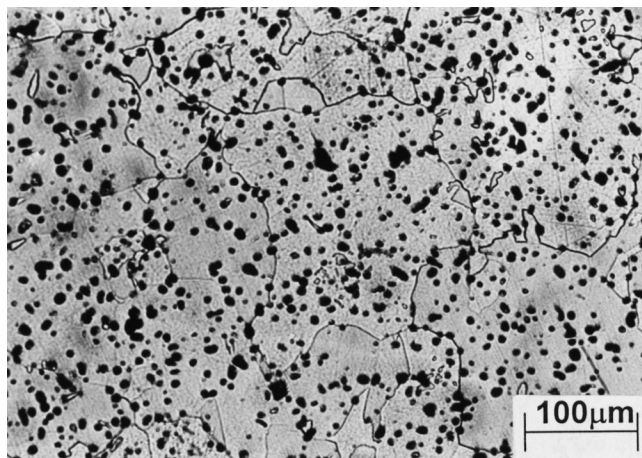
One of the benefits of adding inert dispersoids into iron compacts is to retard the grain growth, so that the pores will remain attached to the grain boundaries and, thus, improve densification. The dispersoids, however, could block the diffusion path of iron atoms and interfere with the mass flow of iron atoms, particularly on the grain-boundary diffusion mechanism. The final sintered density is, thus, influenced by these two counteracting effects: grain-boundary pinning and diffusion blocking. Since the grain growth in pure iron compacts was not significant in the α phase, as shown in Figure 7(a), the benefit of retarding the grain growth by adding alumina was not apparent below 912 °C. However, the diffusion-blocking effect was still effective. Thus, sintering in the α phase was inhibited when dispersoids were



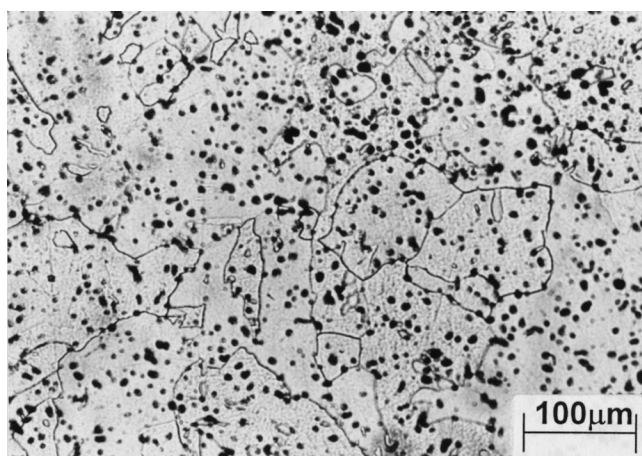
(c)

Fig. 9—The fractured surface, as shown by arrows, of compacts that were heated to 950 °C: (a) pure iron, (b) iron with 0.1 wt pct 0.05 μm alumina, and (c) iron with 0.1 wt pct 0.4 μm alumina.

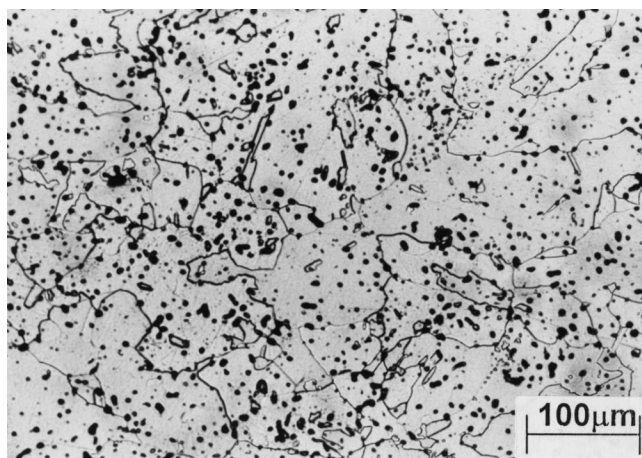
present, as illustrated by the dilatometric curves shown in Figures 5 and 6. As the compact passed the phase-transformation temperature where exaggerated grain growth usually occurs, the grain-boundary pinning effect became distinct, as demonstrated by comparing the microstructures shown in Figures 7(b) and Figure 8(b). These figures also show inhibited neck growth due to the diffusion-blocking effect.



(a)



(b)



(c)

Fig. 10—The microstructure of compacts sintered at 1350 °C for 1 h: (a) pure iron, (b) iron with 0.1 wt pct 0.4 μm alumina, and (c) iron with 0.1 wt pct 0.05 μm alumina.

As more dispersoids were added into the compact, the grain-boundary pinning effect, which is beneficial for densification, became more apparent. However, with the addition of more dispersoids, more diffusion paths are blocked, and the densification is retarded. It is, thus, reasonable to expect

Table II. The Percentage of the Surface Area of a 7 μm Iron Powder, Which Is Covered by Alumina Particles at Various Weight Fractions

Alumina Particle Size, μm	Alumina Content, Wt Pct					
	0.10	0.20	0.50	1.00	1.45	12.7
0.40	0.8	1.6	3.9	7.9	11.4	100
0.05	6.8	13.7	34.1	68.8	100	—

that there is an optimum alumina content at which the grain-boundary pinning effect overshadows the diffusion-blocking effect and results in an improved sintered density. Singh^[10] studied a case in which 7 μm iron powder was completely covered by 0.005 to 0.03 μm aluminum oxides. No densification enhancement was observed, because the grain-boundary diffusion mechanism of iron was completely blocked. The calculated amounts of alumina with which the 7 μm iron powder surfaces were completely covered were 0.11 wt pct for 0.005 μm alumina and 0.86 wt pct for 0.03 μm alumina, respectively. In this study, the amounts of dispersoids needed for such complete surface coverage were 1.45 wt pct and 12.7 wt pct for the 0.05 and 0.40 μm alumina powders, respectively. Thus, should there be a density improvement, the optimum amount of alumina should be less than these two quantities. The results of this study showed that, at 1350 °C, sintering was improved by adding 0.1 and 0.2 wt pct of 0.05 μm alumina particles. These two alumina quantities gave only 6.8 and 13.7 pct coverage on the iron powder surface, respectively. The percentages of the surface area of a 7 μm iron powder covered by alumina of other contents employed in this study are listed in Table II.

Zener's theory^[18] suggests that finer alumina particles result in a stronger pinning effect and less grain growth. This implies that a higher sintered density might be obtained by using finer alumina. In reviewing previous literature, which addressed the effect of fine dispersoids on the sintering of carbonyl iron powders, it is noted that Singh and Houseman^[9] used 0.005 to 0.03 μm alumina and that the minimum amount was 0.5 wt pct. Assuming that the additives were uniformly distributed on the iron powder surface, this would mean that all iron powder surfaces were covered when 0.005 μm alumina was used. For 0.03 μm alumina additions, 58 pct of the surface area was covered. Corti and Cotterill^[8] used alumina particles smaller than 0.03 μm , and the minimum amount employed was about 0.16 wt pct. This implies that more than 19 pct of the iron powder surface was covered. It is very likely that too much alumina was used in these studies, and, thus, no enhanced sintering was observed.

Another factor that may explain why previous studies did not find improved densification is the different types of carbonyl iron powders used. The frequently used types contain about 0.6 to 0.8 wt pct carbon, which is much higher than the 0.065 pct of the reduced grade used in this study. Some others contain a small amount of silica, which was used as a tumbling media during powder preparation.^[19] Since both carbon and silica could inhibit the exaggerated grain growth, the beneficial effect of adding alumina could, thus, be overshadowed.

In a previous study by Lu *et al.*,^[14] it was found that an aluminum stearate addition improved the densification of

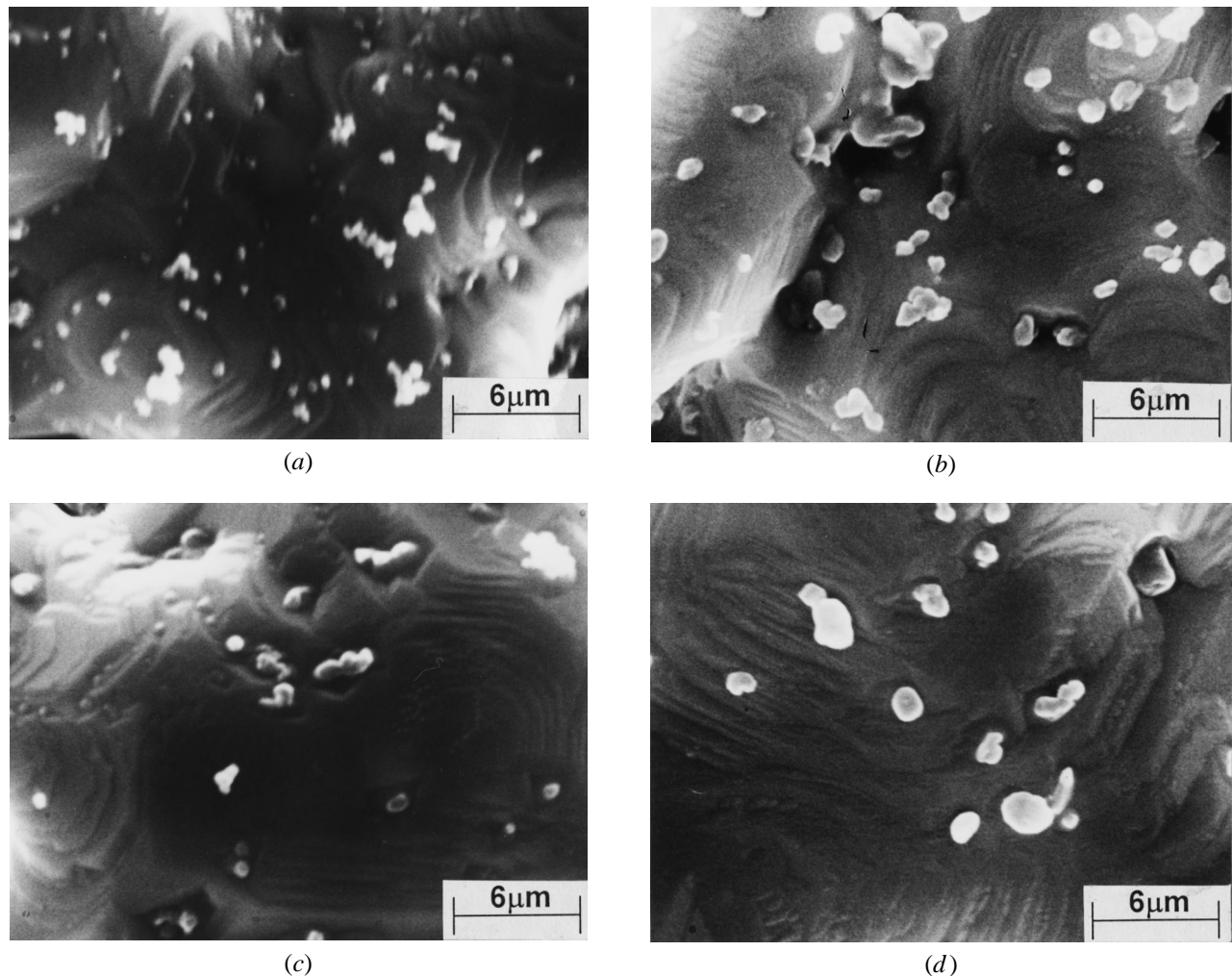


Fig. 11—The morphology changes of alumina particles at different temperatures: (a) 1200 °C, 0.05 μm alumina; (b) 1200 °C, 0.40 μm alumina; (c) 1350 °C, 0.05 μm alumina; and (d) 1350 °C, 0.40 μm alumina.

Table III. The Effects of 0.1 Wt Pct 0.4 μm and 0.05 μm Alumina on the Sintering Behavior of Carbonyl Iron Powder Compacts

Compact Type	α -Phase Region		γ -Phase Region				Final Sintered Density
	Inhibition Effect	Degree of Sintering	Pore Isolation from Grain Boundary	Degree of Sintering	Alumina Coarsening	Final Grain Size	
Pure iron	o	o	o	o	o	o	o
With 0.4 μm alumina	+	-	-	+	+	-	-
With 0.05 μm alumina	++	--	--	++	++	--	+

o: the status of pure iron; +: increased; ++: significantly increased; -: decreased; and --: significantly decreased.

carbonyl iron compacts that were prepared by both the powder injection molding and the die-compaction techniques. Their results, from inductively coupled plasma analysis, indicated that some aluminum in the stearate was dissolved in the iron matrix, while some other aluminum formed alumina during debinding and sintering. Both of these phenomena were attributed to the enhanced sintering; notably, however, no experiment was performed to identify the contribution of each individual effect. This study shows that fine

alumina particles alone can improve the densification of carbonyl iron compacts.

Another factor that affects the sintering behavior of iron compacts is the coarsening of fine alumina particles. Figure 11 illustrates that, at high temperatures, coarsening of alumina occurred, particularly on the 0.05 μm particles. This reduces the blocking effect and contributes to the improved densification at high temperatures.

One of the benefits of adding fine oxides to metal powders

has been to retard the normal grain growth of the metal matrix during the final stage of sintering. However, for iron, it provides an additional benefit of inhibiting the exaggerated grain growth during the phase transformation at 912 °C. Thus, it is worthwhile to find out which effect contributes more to the final enhanced densification. Another experiment was, thus, carried out by adding the same 0.05 μm alumina particles to carbonyl nickel powders, which have no allotropic transformation during heating. Although grain refinement was observed, no sintering improvement was found at either 1200 °C or 1350 °C. This suggests that the main role of dispersoids in iron is not in inhibiting the normal grain growth, but more in retarding the exaggerated grain growth during the phase transformation.

V. CONCLUSION

1. In contrast to previous reports, the addition of 0.05 μm alumina to carbonyl iron powders increased the sintered density when compacts were sintered at 1200 °C and 1350 °C for 1 hour in hydrogen. When larger-sized 0.4 μm alumina was employed, no improvement in density was found.
2. Dilatometric analysis showed that fine 0.05 μm alumina hindered the densification of iron compacts in the early stage of sintering. However, it inhibited the exaggerated grain growth during the phase transformation and helped densification at high temperatures.
3. The main contribution of adding fine alumina particles is in inhibiting the exaggerated grain growth for iron during the phase transformation, not in retarding the regular grain growth, which usually occurs at the final stage of the sintering. When carbonyl iron powder was replaced by carbonyl nickel powder, which has no allotropic transformation during heating, no sintering improvement was observed.
4. The wet mixing technique gives a more-uniform alumina distribution and results in a higher sintered density than that obtained by using the dry ball-milling method.

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