

# Improved Dimensional Control of PIM Compacts Through the Adjustment in Sintering Parameters

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## ABSTRACT

The low solid content in powder injection molded (PIM) parts usually causes large amount of shrinkage after sintering and thus poor dimensional stability. This has become one of the main weaknesses for PIM to compete with other manufacturing processes. To improve the dimensional control, the effect of sintering temperature, heating rate, and the phase transformation that occurs during sintering were investigated. The results show that when abrupt dimensional change occurs, such as during the  $\alpha \rightarrow \gamma$  phase transformation, or when high shrinkage rate occurs, the dimensional stability deteriorates. By slowing down the heating rate in the region where high shrinkage rate occurs, avoiding the phase changes, and adding alloying elements to broaden the phase transformation temperature range, the dimensional control of PIM compacts can be improved.

## INTRODUCTION

The major advantages of powder injection molding (PIM) process are its capability in fabricating small and complex-shaped parts with high density, good mechanical properties, and surface finishes. However, one of the disadvantages of this technology is its poor dimensional stability[1]. This is because fine powders and low solid contents are used in the feedstock and cause large amount of shrinkage during sintering.

Dimensional changes can be observed in at least three stages of the PIM processes. The first stage occurs just after the molding when the material is cooled to the ambient temperature in the mold cavity. The next stage of shrinkage occurs during debinding. Lin and Hwang used a laser dilatometer to observe the dimensional changes during solvent debinding. Their results show that the binder swells when the parts are immersed in the solvent. When the binder amount, binder type, debinding temperature, and solvent type are not selected carefully, distortions and inconsistent dimensional changes will occur[2]. Previous studies on thermal debinding also show that abrupt shrinkages and expansions of the compacts occur during thermal debinding and impair the dimensional stability. Slower heating rate, smaller amount of backbone binders, and inert gases or vacuum for the debinding atmosphere help alleviate the problem[3].

The major dimensional change in the PIM parts occurs in the last stage, the sintering process. German indicates that high sintering temperature and fast heating rate cause poor dimensional stability due to the anisotropic shrinkages[4]. Bulger and Erickson also postulate several causes of the dimensional problem: (1) gravity effect, (2) low solid content, and (3) non-uniform green density.[5-7] Other studies indicate that

large parts and coarse powders are more likely to have larger amount of distortions than do small parts and fine powders[8,9].

The purpose of this study is to further investigate the effect of sintering parameters on the dimensional changes of PIM parts. With an understanding of the causes of these dimensional changes, the distortion and tolerance control of PIM parts can be improved by adjusting processing parameters.

## EXPERIMENTAL PROCEDURE

The fine carbonyl iron powder was selected in this study as the base powder. The characteristics of the powder are given in Table I. To understand the effect of alloying elements on the tolerance control, Ni and Fe<sub>3</sub>P, which are austenite and ferrite stabilizers, respectively, were added into the iron powder. To prepare the feedstock, the iron powder was mixed with a wax-based binder. After kneading, the feedstock was molded into rectangular specimens of 2 x 10x 100mm.

A two stage debinding process was employed. For solvent debinding, the compact was immersed in heptane until about 80% of the soluble binders were removed. During the subsequently thermal debinding, the solvent debound compacts were heated at 5 °C/min to 650 °C, holding for 1 hour in a hydrogen atmosphere to remove the remaining binder.

Sintering was carried out under hydrogen. To attain the same 95% relative density so that the dimensional stability can be compared on the same basis, the sintering temperature and time were adjusted for each group of the specimens. The data reported are averages of a minimum of eight compacts.

To understand the effect of sintering parameters on the dimensional stability of PIM parts, a thermal dilatometer was employed to monitor the in-situ dimensional changes of specimens. The dilatometer analysis was carried out using the same parameters as those employed for the sintering runs.

Table I. The characteristics of the carbonyl iron powder was used in this study

Iron Powder	Characteristics
Type	Carbonyl Iron Powder
Designation	CIP-S-1641
Average Particle Size (Laser Scattering Method)	D(10) 2.05µm D(50) 4.24µm D(90) 8.81µm
Shape	Spherical
Density (Pycnometer)	7.57 g/cm <sup>3</sup>
C, wt%	0.741
O, wt%	0.812
Supplier	ISP

## RESULTS

### Effect of Heating Rate

Figure 1 shows that the dimensional controls of injection molded specimens were good after molding, solvent debinding, and thermal debinding. However, the standard deviation increased significantly after

sintering due to the large amount of shrinkage. Among the three heating rates employed, 5, 10, and 20 /min, the slowest heating rate of 5 /min produced the best result. To understand such differences caused by the heating rates, dilatometry analysis was performed. Figure 2 shows the amount of shrinkage during heating increased as the heating rate decreased. All three curves deflected at about 912 when  $\alpha \rightarrow \gamma$  phase transformation occurred. The shrinkage then slowed down significantly due to the decreased diffusion rate and the exaggerated grain growth that accompanied with the phase changes[10]. When the shrinkage rates were measured as illustrated in Figure 3, a large amount of abrupt change was observed at the phase transformation, particularly when the heating rate of 20 /min was employed. It was believed that the abrupt dimensional change at 912 caused the deteriorated dimensional control. To confirm that, a group of specimens were sintered below the phase transformation temperature at 900 .

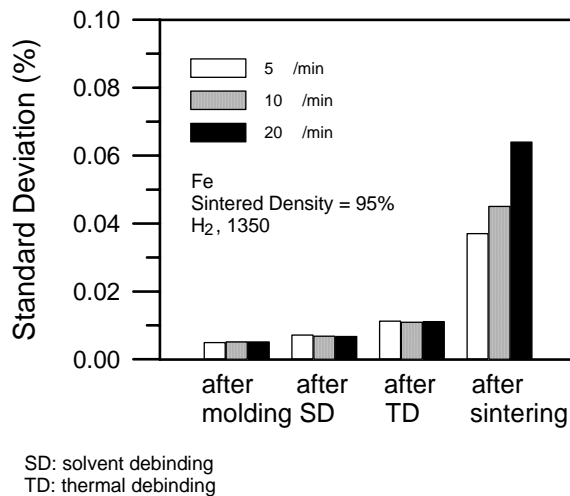


Figure 1. The effect of heating rate on the standard deviation of the length of PIM iron compacts.

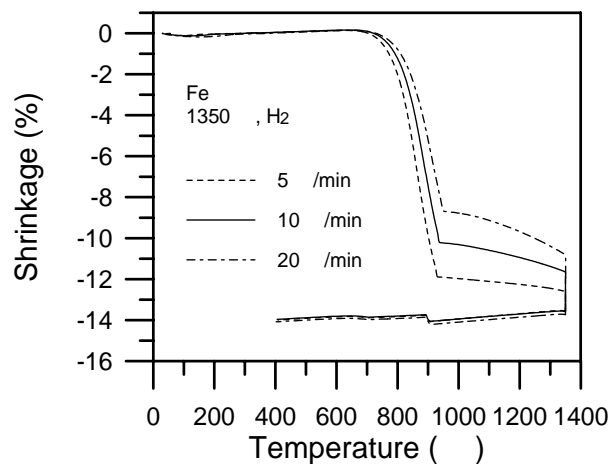


Figure 2. The dilatometer curves of iron compacts heated at 5, 10, 20 /min, respectively, to 1350 in hydrogen.

The holding time was 45 minutes so that the same density of 95% was attained as that sintered at 1350 for 2.5 hours. The short sintering time of 45 minutes, compared to the 2.5 hours required for 1350 sintering, confirmed that sintering in the  $\alpha$  phase and the avoidance of the exaggerated grain growth enhanced the densification. The differences in the standard deviation between specimens sintered at these

two temperatures suggested that the sudden volume change, which amounts to 1.5%, is the main cause of the deteriorated dimensional control.

Another group of specimens, which were sintered to 95% density at 900 °C for 45 minutes, were heated to 920 °C right after sintering and then immediately furnace cooled. The density increase was less than 0.5%. The standard deviation, as shown in Figure 4, was almost the same compared to that sintered

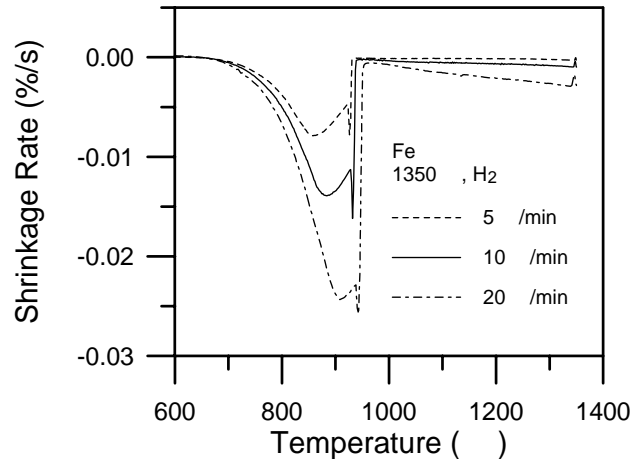


Figure 3. The shrinkage rates of iron compacts heated at 5, 10, 20 °C/min, respectively, to 1350 °C in hydrogen.

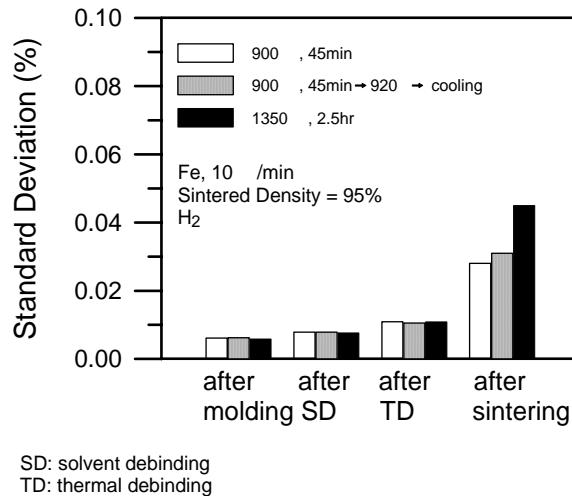


Figure 4. Standard deviations of the lengths of PIM iron compacts that were sintered in different sintering conditions.

isothermally at 900 °C without further heating. These results suggest that the phase transformation does not affect the dimensional stability when the compact is already dense. The phase transformation, however, could impair the consistency of the specimen length when it occurs on low density parts, such as when high heating rate is used.

### Effect of Alloying

The above study shows the significant effect of the heating rate on the dimensional control of sintered

PIM iron compacts. To improve the dimensional stability, the compact can be sintered below 912 °C to avoid the phase transformation or by slowing down the heating rate so that the compact is relatively dense when the phase transformation occurs. These methods, however, only apply for pure iron, which are frequently used for soft magnets. For most other applications, such as in structural parts, the sintering behavior is quite different due to the alloying elements present. To understand the effect of the alloying elements, Fe-0.7wt%P and Fe-8wt%Ni were employed.

Figure 5 shows that when 0.7%P was added, the shrinkage curve became very smooth, without any deflection at 912 °C. This indicated that most phosphorous was dissolved into the matrix and the whole sintering cycle was carried out in the  $\alpha$  phase. For Fe-8%Ni, the phase transformation was noticed at a lower temperature and the deflection was not significant. This is because nickel has a slower diffusion rate than phosphorous and thus the nickel was much less homogenized during heating. This non-uniform nickel distribution caused the phase transformation to occur continuously at different locations of the compact and in the range between 912 °C and 750 °C, which is the phase transformation temperature of Fe-8%Ni[11,12]. Since the phase transformation occurred in a wide temperature range, the shrinkage rate that was caused by the volume change between the  $\alpha$  and the  $\gamma$  phases decreased. Thus, as shown in Figure 6, much less abrupt change on the shrinkage rate curve was noticed during heating compared to that of the pure iron compact.

The effects of Ni and P additions on the dimensional stability are shown in Figure 7. The results illustrate that when there was no phase transformation, such as in the case of Fe-0.7P, the dimensional stability was the best. The Fe-8%Ni compact also show improved results compared to that of the pure iron. This is because the phase transformation, though still noticeable, occurred in a continuous mode and abrupt dimensional changes were avoided.

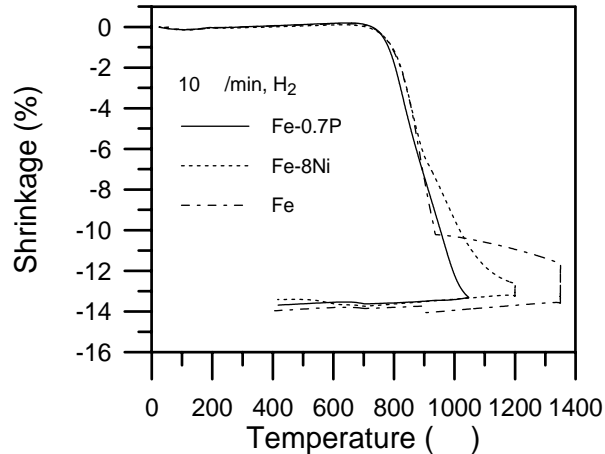


Figure 5. The dilatometer curves of Fe, Fe-0.7wt%P, and Fe-8wt%Ni compacts heated at 10 °C/min in hydrogen.

## DISCUSSION

The previous results show that, when the phase transformation occurs during sintering, the dimensional stability deteriorates significantly. This is because the compact shrinks suddenly during the phase transformation, particularly when the compact is only loosely sintered and the interparticle bonding is still weak. Since the volume decrease is quite large at about 1.4% when the  $\alpha$  phase transforms into the  $\gamma$

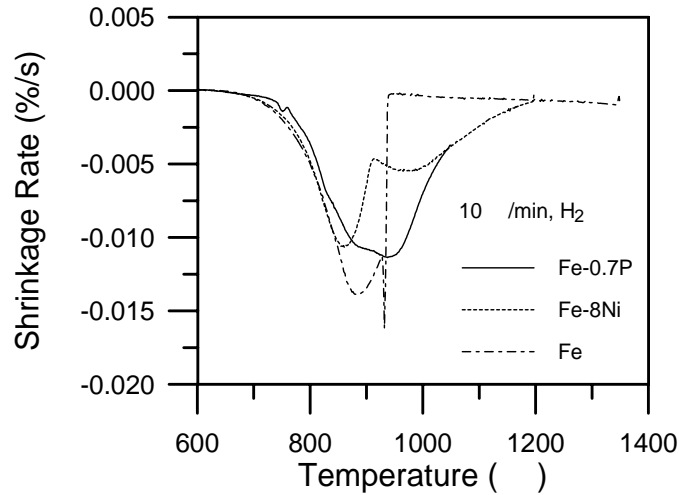


Figure 6. The shrinkage rates of Fe, Fe-0.7wt%P, and Fe-8wt%Ni compacts heated at 10 °C/min in hydrogen.

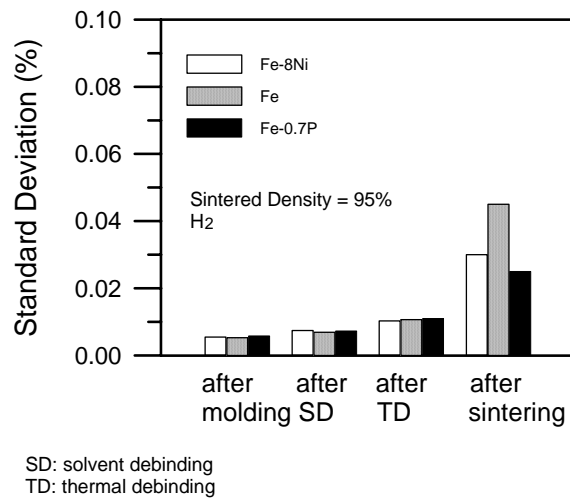


Figure 7. The effect of Ni and P additions on the standard deviation of the length of PIM compacts.

phase, this sudden volume change could cause loosely bonded particles to be pulled or pushed around. It is very likely that these local deformations could cause uneven shrinkages. Since the composition and the temperature are not perfectly uniform within the compact, an isotropic and even deformation is not likely. Thus, the dimensional stability becomes deteriorated.

This is confirmed in Figure 4, which shows that, when the compact has already attained high density and with strong interparticle contacts before reaching 912 °C, the phase transformation has little effect on the dimensional stability. Figure 7 also shows that when the  $\alpha$  phase stabilizers, such as phosphorus, are added, the phase transformation of iron is eliminated and thus the dimensional stability is improved. When nickel is added into iron powders, the sintering curve is also smooth. However, this is not due to the elimination of the phase transformation, but, because the phase transformation occurs in a wide temperature range. Since the abrupt length change is prevented, the dimensional control is also improved.

With the understanding of the effects of heating rates and phase transformations on the dimensional

stability, an experiment was carried out to illustrate the benefits of adjusting these two factors. The specimens used were Fe-0.7%P. As shown in Figure 8, the heating rate of the this group was adjusted to 5 /min between 700 and 1100 so that its maximum shrinkage rate was only half of that attained by the first group, which was a heating rate of 10 /min for the whole heating stage. With the elimination of the phase transformation by adding 0.7% P and the reduced shrinkage rate, the dimensional stability of the second group was improved, as illustrated in Table II.

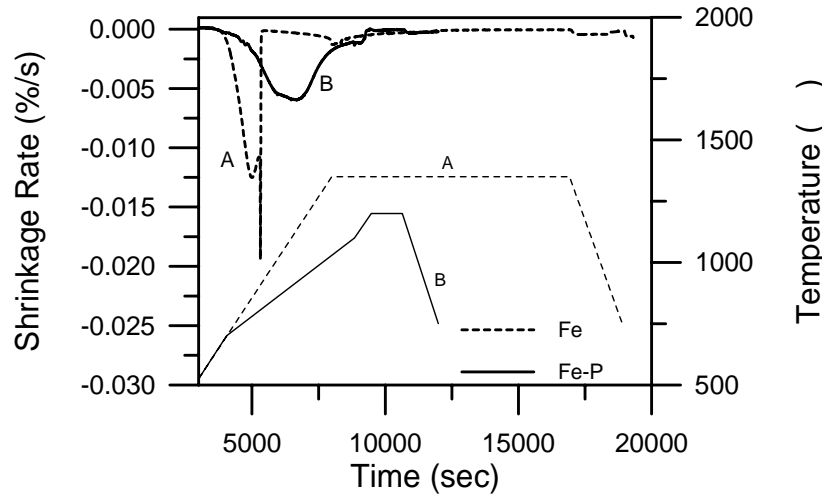


Figure 8. The shrinkage rate of Fe and Fe-0.7P PIM compacts with different sintering conditions.

Table II. The amount of shrinkage and the standard deviation of Fe and Fe-0.7P PIM compacts with different sintering parameters

	Fe	Fe-0.7P
Shrinkage (%)	14.08	14.12
Standard Deviation (%)	0.045	0.021

## CONCLUSIONS

This study shows that the dimensional stability of PIM iron compacts is influenced by the occurrence of the phase transformation and the heating rate. When phase transformation occurs, the large volume change could cause poor dimensional control. This becomes worse when a high heating rate is employed since the density of the compact is low and the interparticle bonding is weak when the phase transformation occurs. By adding phosphorous, which is a  $\alpha$  phase stabilizer, and using a low heating rate, the standard deviation of the length of the iron compact improves from 0.045 to 0.021%. When Ni is added, the phase transformation occurs in a wide temperature range and alleviates the problem of sudden volume changes. Thus, the dimensional stability also improves. Good dimensional stability can be attained by selecting adequate alloying elements and with adjusted heating schedules.

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powder.

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