



FACTORS AFFECTING THE DIMENSIONAL CHANGE OF SINTER HARDENING POWDER GRADES

by

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ABSTRACT

The use of sinter-hardening steel powders combined with furnaces equipped with fast cooling units is a cost effective process allowing the production of parts with high strength and apparent hardness without the use of subsequent heat-treatment. However, the high hardness achieved after sintering makes subsequent machining or sizing operations very difficult. Therefore, it becomes crucial to carefully control the final dimensions of parts.

The use of a dilatometer is a powerful tool to better understand the phenomena that occur during sintering and affect the final dimensions of parts. In particular, dilatometry can be used to develop powder mix formulations with an improved dimensional stability. This paper presents the results of dilatometric tests carried out on sinter hardening powders admixed with different types and levels of additives. In particular, the influence of graphite, copper and lubricant grades on the dimensional variation during sintering is presented. The effects of sintering temperature and time on the final dimensions are also discussed.

INTRODUCTION

As powder technology takes over a larger part of the market from other technologies, the requirements of new applications become more and more demanding. In order to fully respond to the more challenging applications at a competitive cost, high performance sinter-hardening grades are used. However, the high apparent hardness grades require a deep understanding of every factor susceptible to influence their dimensional change during sintering since it is becoming very difficult to adjust final dimensions by secondary operations like machining or sizing. Dilatometry is a very powerful tool to better understand phenomena occurring during sintering and thus develop more robust materials or formulations and/or improve process conditions. Dilatometry was used by many authors in the past to elucidate the effect on dimensional change during sintering of various factors, such as heating and cooling rates [1], atmosphere composition [2], base powder composition and residual impurities like sulfur [2, 3],

oxygen [4], silicon [5], and lubricant chemistry [6, 7]. The effect of density and size distribution of the base powder and/or the additives has also been studied [8, 9]. Complexity of interactions between additives makes establishing general rules very difficult in regard to the optimum choice of additives. Dilatometric studies have allowed optimization of premix composition containing graphite, copper and nickel relative to their dimensional stability [10].

A dilatometry study was carried out by the authors to better understand and quantify the effect of different factors that could induce variation of the dimensional change during sintering of two different sinter-hardening steel powder grades, ATOMET 4601 and 4701. The influence of levels of graphite and addition of copper was investigated. Particular attention was paid in the study to the influence of the nature and size distribution of graphite on the dimensional behaviour of specimens sintered in a dilatometer. Indeed, there are many different grades of graphite on the market. One can choose between natural or artificial graphite. Graphites of different sizes are also available. For most applications, natural graphite is widely used because of their low prices. However, natural grades typically contain from 3 to 5% ash, which varies in chemical composition, mainly because of the source. Artificial or synthetic graphites are slightly more expensive but show a much higher level of purity and contain nearly 100% active carbon. It is reported that carbon diffuses in the iron matrix at a lower temperature with synthetic graphite [11]. Of particular attention was the influence of carbon diffusion on the growth caused by the copper melting at about 1085°C. Finally, the influence of internal lubricant type and heating rate was also examined.

EXPERIMENTAL PROCEDURE

I. Mixes and specimen preparation

Lab-scale mixes with various levels of graphite and copper were prepared with two different sinter-hardening steel powder grades, ATOMET 4601 and 4701. Table 1 gives a brief description of these two powder grades produced by Quebec Metal Powders Limited as well as the mix formulations prepared in that study. Wax was used as internal lubricant for most of the mixes. Mixes with Zn stearate and others without lubricant were also prepared in order to better understand the influence of lubricant on the behaviour of specimens. Different grades of graphite were used to better understand the influence of the nature and size distribution of graphite on the diffusion of carbon in the iron lattice and the dimensional behaviour of specimens, in particular, the growth caused by the copper melting. Table 2 gives a brief description of the different graphite grades evaluated in the study.

Table 1. Characteristics of the base powders used in the study and the pre-mixes formulation.

Steel Powder	Hardenability *	Pre-alloyed elements, wt%					Admixed additives, wt%		
		<i>Ni</i>	<i>Mo</i>	<i>Mn</i>	<i>Cr</i>	<i>O₂</i>	<i>Cu</i>	<i>Graphite</i>	<i>Lubricant</i>
ATOMET 4601	8.4	1.8	0.55	0.2	0.05	0.10	0 or 2	0, 0.6, 0.9 or 1.0	0.75
ATOMET 4701	26	0.9	1.0	0.5	0.5	0.20	2	0.9	1.0

* *Hardenability is estimated by multiplying the hardenability factor of each alloying elements [12]*

Table 2. Characteristics of different grades of graphite used in the study.

	Type	Ash content, %	Moisture, %	D50, μm	D90, μm
A- SW1651	Natural-Reg	3-5	0.5	9-10	20-25
B- 3203S	Natural-Fine	3-5	0.5	6	10
C- SW1645	Natural-Coarse	3-5	0.5		
D- F25	Synthetic-Reg	<0.1	0.1	10	25
E- F10 or KS10	Synthetic-Fine	<0.1	0.1	6	10

II. Description of dilatometric tests

Two different types of specimens were prepared for dilatometry, standard TRS or dog bone specimens. Both specimens were pressed on a hydraulic press to a density of 6.8 g/cm^3 . In the case of mixtures containing no lubricant, compaction was done with the die wall lubrication technique by spraying a Zn stearate lubricant on the die walls. Specimens were treated at 600°C to burn-off the lubricant prior to the dilatometry test. After delubing and prior to testing, the specimens were cut at one end in the case of TRS specimen and in the middle portion of the specimen in the case of dog bones to obtain a specimen 1 inch long.

Dilatometry tests were carried out in a theta dilatometer, Dilatronic 1600°C . Specimens were heated at a rate of $10^\circ\text{C}/\text{min}$, maintained 40 min at $\sim 1120^\circ\text{C}$ and cooled at an initial rate of $10^\circ\text{C}/\text{min}$ under an atmosphere of pure Argon or Argon with 5% H_2 . A few specimens were also heated at a rate of $20^\circ\text{C}/\text{min}$ to evaluate the effect of time on expansion behaviour. Figure 1 shows the typical temperature profiles applied to the specimens. It should be noted that the dilatometer used could not maintain the cooling rate constant during the test. The cooling rate in the $650\text{-}400^\circ\text{C}$ range was $6^\circ\text{C}/\text{min}$ ($11^\circ\text{F}/\text{min}$) or $0.1^\circ\text{C}/\text{sec}$ ($0.2^\circ\text{F}/\text{sec}$). This is significantly lower as compared to the cooling rate achieved in industrial sintering furnaces. A cooling rate up to $2^\circ\text{C}/\text{sec}$ can be achieved in sintering furnaces equipped with fast cooling units.

Raw data for each test was processed with an MSEXcel spreadsheet allowing the calculation of the specimen dimensional change, throughout the duration of the test, the start and end temperatures, as well as the amplitudes (contraction or growth) of the major phenomena occurring during sintering: the $\alpha \rightarrow \gamma$ and $\gamma \rightarrow \alpha + \text{carbides}$ phase transformations, graphite diffusion, copper melting and growth, contraction at isotherm and linear expansion coefficient of the α and γ phases. In order to allow a direct comparison between the tests, the specimen expansion was measured in percentage of variation as compared to the

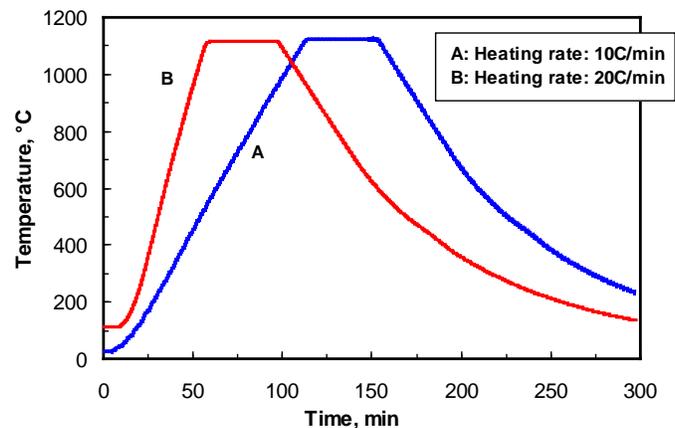


Figure 1. Temperature profile used for dilatometry.

size of the specimen at 200°C during the heating cycle. As a result, readers will note that the dimensional change of all the curves shown in this paper is equal to 0 at 200°C. Also, for that reason, the overall dimensional change that a part overcame during the test was estimated when the temperature of the specimen reached 200°C during cooling. In this paper, dilatometry profiles are illustrated by the dimensional change of the specimen as a function of the temperature as shown in Figure 2 for a FLC-4608 specimen. The key changes are illustrated along with a legend.

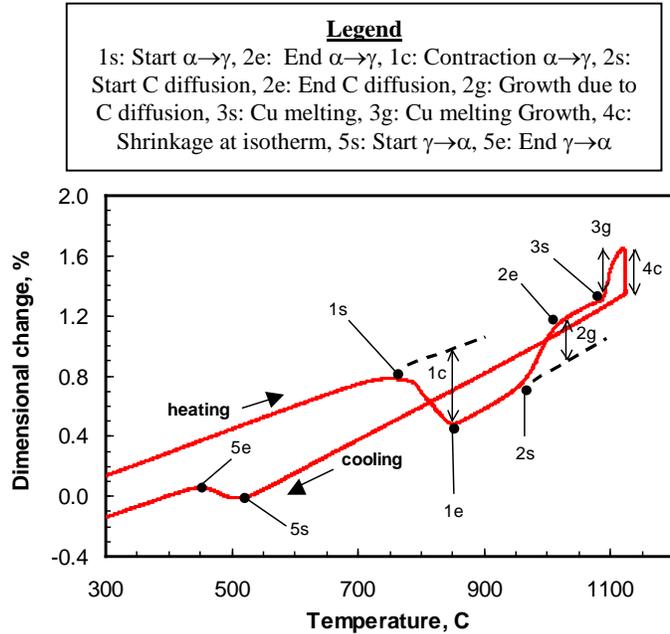


Figure 2. Typical dilatometric profile of FLC-4608 specimen (AT4601-0.9% graphite- 2%Cu).

RESULTS AND DISCUSSION

I. Effect of graphite and copper addition

Figure 3 shows typical dilatometric profiles of ATOMET 4601 pressed to 6.8 gcm³ and containing various levels of graphite: (A) no graphite, (B) 0.6% graphite, (C) 0.9% graphite. All the specimens show very similar expansion up to 700°C. The expansion rate was around 0.0014%/°C between 200 and 300°C and 0.00148%/°C between 500 and 700°C, which closely corresponds to the well to the thermal expansion coefficients of ferrite. Specimens start to shrink above 750°C. This corresponds to the $\alpha \rightarrow \gamma$ phase transformation. The $\alpha \rightarrow \gamma$ transformation starts at around 835°C for the specimen with no graphite and is completed at about 875°C. Addition of graphite significantly reduces the $\alpha \rightarrow \gamma$ transformation start temperature, from 835°C to 770°C. The temperature range for which the transformation took place is also almost doubled with graphite, ~80°C versus ~40°C. This is explained by the diffusion of a very small quantity of carbon in the iron lattice prior to the start of the transformation [13]. It should be noted that adding more graphite does not affect the $\alpha \rightarrow \gamma$ start and end temperatures.

After completion of the $\alpha \rightarrow \gamma$ phase transformation, the specimens expand again with rising temperature. The expansion rate is slightly higher between 800 and 950°C for specimens with graphite as shown in Figure 4. This is due to the diffusion of a slight amount of graphite in the γ lattice. However, the diffusion of carbon in the γ phase really accelerates at about 950-970°C for these specimens as indicated by the increase in expansion rate, Fig. 4. The carbon diffusion ends at about 1020 to 1040°C when expansion rate decreases and stabilizes again. It can be seen that the expansion rate, as well as the total expansion associated with the carbon diffusion, are proportional to the level of graphite in the mix. Also, increasing the level of graphite led to a slight reduction of the temperature at which the diffusion of carbon was completed. At

temperatures above 1050°C, the expansion rate decreases for all the specimens as shown in Figure 4. In fact, the expansion rate between 1020 and 1120°C is significantly lower than the expansion coefficient of γ during cooling. This is explained by the diffusion of iron atoms that likely begin to cause the shrinkage of the specimens at about 1000°C. It should be noted that the shrinkage is more pronounced in specimens containing carbon, confirming that this element activates the diffusion and sintering processes.

For all the specimens, significant shrinkage is obtained when specimens are maintained at the maximum temperature. The shrinkage measured was about 0.30% after 40 minutes at ~1120°C. During cooling, specimens shrink at a rate of about 0.0022 to 0.0025%/°C, depending on temperature. The $\gamma \rightarrow \alpha$ (+carbides) transformation starts at 790°C for the specimen without graphite and at 560 and 545°C for specimens with 0.6 and 0.9% graphite respectively. The growth associated with this phase transformation decreases also with the level of carbon, from 0.43 to 0.25%.

Figure 5 shows the effect of copper addition on the expansion profile of FL-4608 formulations. Profiles A and B correspond to ATOMET 4601 with 0.9% graphite, 0 and 2% copper added respectively. Profile C is obtained for a specimen with 1% graphite and 2% copper. Adding copper has no significant influence on the $\alpha \rightarrow \gamma$ transformation and the carbon diffusion. More precisely, copper addition only slightly inhibits the diffusion of graphite below 950°C. All the specimens show almost identical dimensional change between 1020-1080°C. Increasing the graphite from 0.9 to 1% has no influence on the growth caused by the carbon diffusion. The growth associated with the copper melting, which occurred at 1080-1085°C, can be easily observed for both specimens containing copper. Also increasing the graphite content from 0.9 to 1% led to a significant reduction of the growth associated with the melting and penetration of copper in the iron skeleton, from 0.31% to 0.20%. It should be noted that the difference in copper growth as observed by dilatometry between 0.9 and 1% graphite matches the difference in dimensional

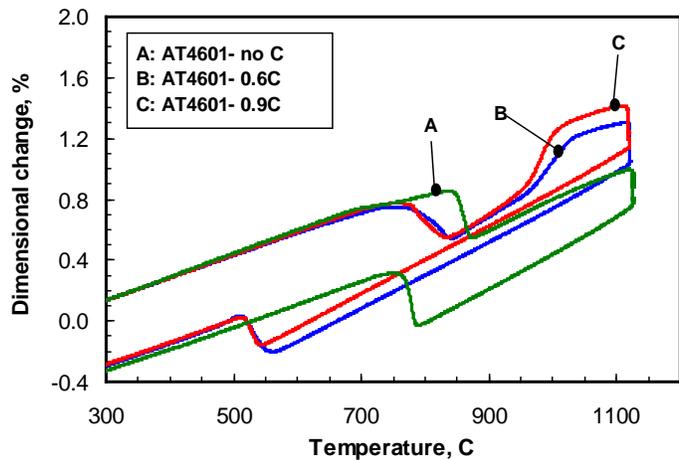


Figure 3. Dilatometric profiles of ATOMET 4601 with various levels of graphite (A) 0%C (B) 0.6%C (C) 0.9%C.

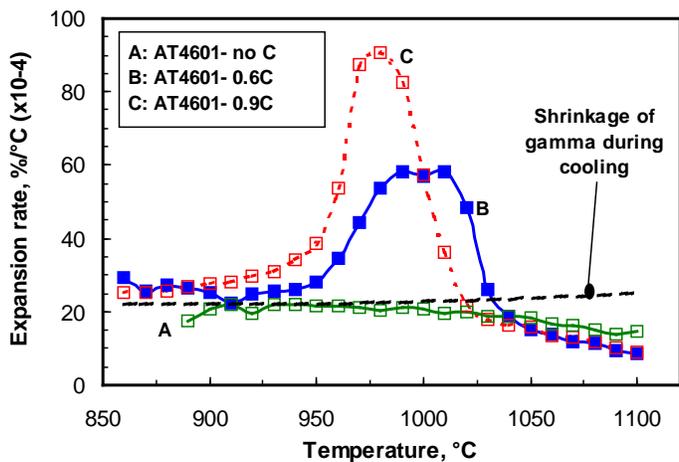


Figure 4. Expansion rate in the γ phase as a function of temperature of ATOMET 4601 with various levels of graphite: (A) No C, (B) 0.6%C (C) 0.9%C.

change obtained under normal sintering conditions. Finally, adding copper did not affect the coefficient of expansion of α and γ . However, it affects the transformation $\gamma \rightarrow \alpha$ (+carbides) slightly by lowering the transformation start and end temperatures and the growth associated with that transformation.

II. Effect of graphite type

At first, the effect of graphite type was evaluated with a FL-4605 formulation made with ATOMET 4601. The influence of graphite grade was also evaluated with a FLC-4608 formulation using ATOMET 4601 and 4701.

a. Effect of graphite type for ATOMET4601 mixes without copper

Figure 6 shows the dilatometric profiles of FL-4605 specimens containing a natural and a synthetic graphite having similar size distribution, A and D respectively, while Figure 7 shows the profiles of specimens containing natural and synthetic graphite of different size. Figure 8 shows the variation of the specimen expansion rate as a function of the temperature. The size characteristics of graphite are summarized in Table 2. All specimens contained a total of 0.6% graphite. It should be noted that the level of active carbon is about 3 to 5% higher in the case of the synthetic grades, giving slightly higher sintered carbon after the tests. In figure 6, the expansion of specimens is similar up to the $\alpha \rightarrow \gamma$ transformation for both grades of graphite. The $\alpha \rightarrow \gamma$ transformation starts and ends at very similar temperatures. However, the shrinkage is less pronounced with the synthetic graphite D, likely because of the diffusion of a small quantity of carbon in the γ phase during transformation. When the $\alpha \rightarrow \gamma$ is completed, it is seen in Figure 8 that the specimen with synthetic graphite D expands at a much higher rate with temperature than the specimen with graphite A. In fact, the expansion rate of

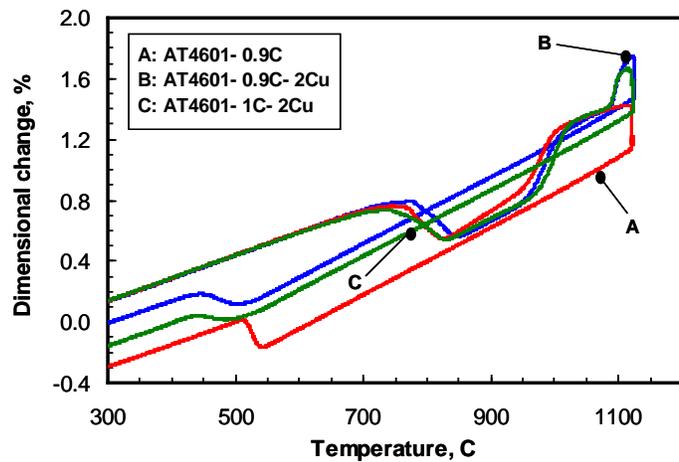


Figure 5. Dilatometric profiles of ATOMET 4601 with graphite and copper: (A) 0.9%C- no Cu, (B) 0.9%C- 2%Cu, (C) 1%C- 2%Cu.

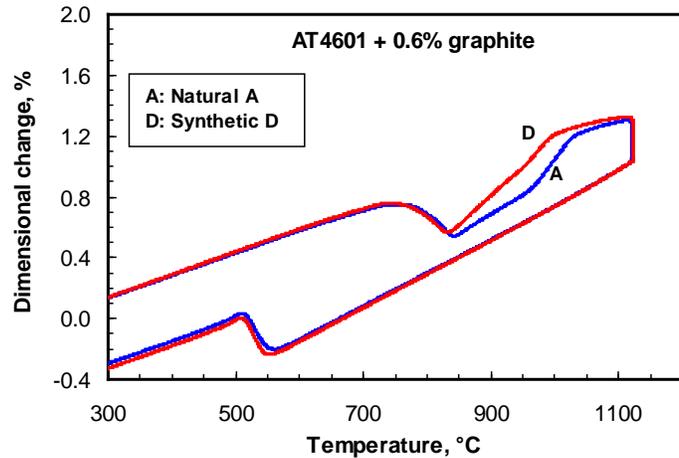


Figure 6. Dilatometric profiles of ATOMET 4601 with two grades of graphite: (A) Natural A, (B) Synthetic D.

that specimen is almost two times higher than the coefficient of expansion of γ during cooling. This is due to the diffusion of carbon that is activated at a much lower temperature with the synthetic graphite, namely around 800-840°C. As explained earlier, the diffusion of carbon in the γ phase is significantly less pronounced with graphite A and really starts to diffuse in the iron lattice at a much higher temperature, 950°C. This result is in agreement with the findings of a study on synthetic graphite [2]. The carbon diffusion is completed at a lower temperature with the synthetic graphite, 1010°C versus 1040°C. The growth associated with the carbon diffusion is also slightly elevated with the synthetic grade. This is explained by the slightly higher level of sintered carbon obtained with the synthetic graphite, namely 0.03% higher. Finally, it can be observed in Figure 8 that the expansion rate of specimen with graphite D is lower between 1020 and 1120°C, indicating that this type of graphite favours the iron diffusion and densification process.

The effect of using finer grades of natural and synthetic graphite on the dilatometric profile is shown in Figure 7. The profiles obtained with graphite A and D are given as reference. All the profiles except the one related to graphite A are very similar. In fact, differences in behaviour between the different grades is better illustrated in Figure 8, which shows the variation of the expansion rate of a specimen as a function of the temperature. In the case of synthetic graphite, no significant difference is obtained by using a finer grade, indicating that the size distribution has only a minute effect on the carbon diffusion for this type. However, in the case of natural graphite A and B, the size distribution has a strong effect on the dilatometric profile. In actual fact, even if the $\alpha \rightarrow \gamma$ transformation starts at the same temperature, it ends at a lower temperature with the finer grade. Also, the shrinkage due to the $\alpha \rightarrow \gamma$ phase transformation is significantly reduced, clearly indicating that carbon diffuses in the newly formed γ phase during that step. The specimen expansion just after the transformation is also more pronounced with the finer grade, indicating that the carbon diffuses at a significant rate in the iron lattice just after the completion of $\alpha \rightarrow \gamma$. However, the carbon diffusion rate drops rapidly and becomes similar to that of the regular grade A between 870 and 950°C. The carbon diffusion starts again at about 950°C and ends at about 1020°C. This two-step diffusion process can be explained by the fact

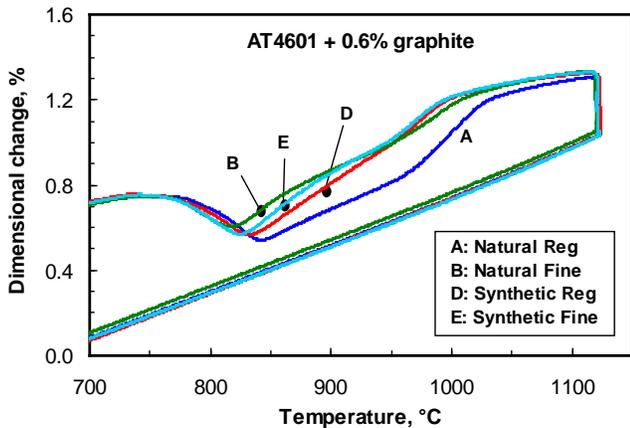


Figure 7. Dilatometric profiles of specimens of ATOMET 4601 with different graphite grades.

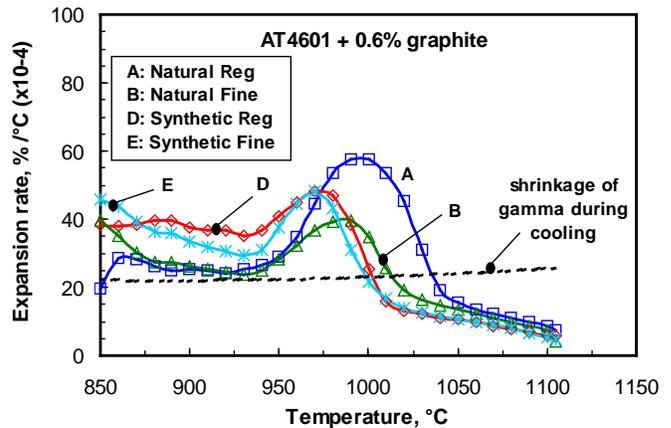


Figure 8. Expansion rate in the γ phase as a function of temperature for specimens with different graphite grades.

that the very fine natural graphite particles diffuse easily while the coarser particles need a higher temperature to diffuse efficiently in the γ phase. This phenomenon is less important with the synthetic graphite grades. Finally, using a finer grade has no real difference on the diffusion process above 1050°C.

In summary, carbon diffuses more easily in the iron lattice if synthetic graphite is used. In addition, size distribution has no influence on carbon diffusivity with synthetic grades while it has a strong effect for natural graphite: the finer the grade, the easier the carbon diffusion.

b. Effect of graphite type for ATOMET 4601 mixes with copper

Previous results clearly showed that using synthetic graphite increased the diffusivity of carbon in the iron lattice. Synthetic graphite also contributed to accelerate the diffusion of iron atoms at temperatures above 1000°C, as indicated by the very low specimen expansion rate measured. It is also known that the level of carbon in the iron lattice affects the Cu growth associated with the melting of this element. In particular, the reduction of the Cu growth, when the carbon level increases, is explained by an increase of the dihedral angle, which inhibits the penetration of Cu into the iron skeleton [14, 15]. Figure 9 shows the influence of the graphite nature on the dilatometric response, and in particular, on the growth caused by the Cu melting for FLC-4608 specimens. The level of graphite in both specimens is 1%. As observed with the FL-4605 formulation, the synthetic graphite D diffuses more easily than the natural one in the iron lattice. The growth due to diffusion of carbon is equivalent for both specimens. Chemical analysis confirmed that the level of sintered carbon was similar, 0.85% for specimen with natural graphite and 0.88% for specimen with synthetic one. A significant difference in growth caused by the melting of copper is observed. In fact, the growth was almost negligible with the synthetic graphite, ~0.04% compared to 0.16% with the natural one. This difference is not due solely to the slight difference in sintered carbon between the two specimens. As a matter of fact, the reduction in Cu growth should have been ~0.03%. The difference is likely due to the superior inter-particle connections due to the higher mobility of iron atoms with the synthetic graphite prior to the melting of copper. The increase in inter-particle bonding inhibits the penetration of copper in the iron skeleton, clearly indicating that the level of sintered carbon is not the only factor affecting the Cu growth for a given powder. According to that result, every change in sintering or material formulation that favours densification and the diffusion process should result in a lower amount of copper penetration between the iron particles, and thus, in lower Cu growth.

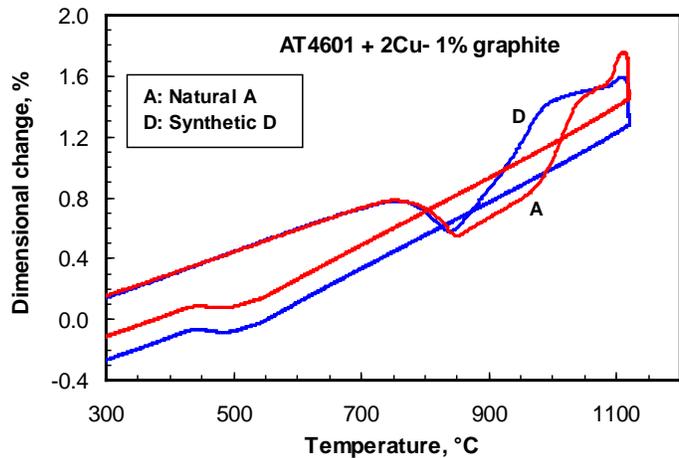


Figure 9. Dilatometric profiles of ATOMET 4601 with copper and natural and synthetic graphite.

c. Effect of graphite type for ATOMET 4701 mixes with copper

ATOMET 4701 is another sinter-hardening grade having a significantly higher hardenability as compared to 4601 powder. Actually, the theoretical hardenability factor of ATOMET 4701 is about 26 as compared to 8.4 for ATOMET 4601. As a result, this grade is used when very high apparent hardness is required, especially for parts with a large volume or when the cooling rate of the sintering furnace is not high enough to sinter-harden other low-alloyed powders. ATOMET 4701 also offers especially good wear resistance due to the very high hardness achieved. Due to its chemical nature, given in Table 1, ATOMET 4701 presents some differences during sintering as compared to ATOMET 4601 as shown in Figure 10 for specimens containing 2% copper and 0.9% graphite A (natural). It should be noted that the level of sintered carbon is slightly lower with ATOMET 4701, around 0.65-0.70% as compared to 0.80% with ATOMET 4601. The $\alpha \rightarrow \gamma$ transformation and the growth associated with the carbon diffusion in the γ phase are shifted to higher temperatures. For example, carbon starts to diffuse at a significant rate at 1000-1020°C as compared to 950°C with ATOMET 4601. The carbon diffusion is still not completed when copper melts, as indicated by the specimen expansion rate, which is above the typical linear expansion rate of austenite. During cooling, the $\gamma \rightarrow \alpha + \text{carbides}$ transformation takes place at a lower temperature range with ATOMET 4701, 470-400°C as compared to 500-450°C with ATOMET 4601. This confirms the higher hardenability of ATOMET 4701.

Figure 11 shows the results of dilatometry tests carried out with ATOMET 4701 powders admixed with copper and different grades of graphite. Graphite grades were selected to obtain very diverse carbon diffusivities in the steel in order to better evaluate the effect of carbon diffusion on the dimensional behaviour of specimens and, in particular, on the growth caused by copper melting. Two natural grades, a regular and a coarse, and one synthetic grade, were selected. It should be noted that the level of copper is 2%, while the level of graphite is 0.9% with the natural grades and 0.88% with the synthetic grade. These changes were made to achieve the same sintered carbon at the end of the test.

As seen in Figure 11, using a coarser grade clearly impedes the carbon diffusion in the γ phase that starts at a temperature of 1030-1050°C. It is also clear that the carbon does not completely diffuse in the austenite and still diffuses at a very significant rate when copper melts. The lower amount of carbon in the iron lattice coupled with an anticipated poor inter-particle connection led to a significant growth when copper melts. In fact, the copper growth is almost two times higher as compared to that obtained with the regular grade. The opposite trend is observed with the synthetic graphite. Conclusively, as discussed in

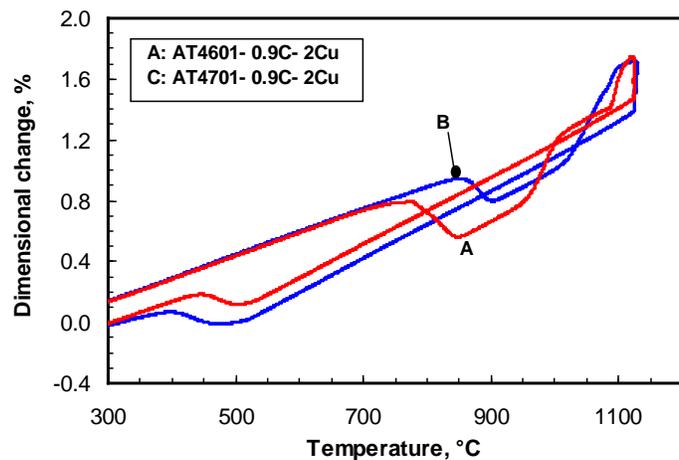


Figure 10. Dilatometric profiles of specimens made with either ATOMET 4601 or ATOMET 4701. (Mix formulation: 0.9% graphite- 2% Cu)

the previous section, the diffusion of carbon in the iron lattice is highly accelerated with synthetic graphite and starts at much lower temperatures. As a result, carbon is completely diffused in the iron lattice and the sintering process is well initiated when copper melts, giving a very low Cu growth.

The influence of carbon diffusion on the copper melting growth is shown in Figure 12 that gives the relation between the growth caused by copper melting and the temperature at which the carbon starts to diffuse in austenite for ATOMET 4601 and 4701 powders admixed with 2% copper and 0.9 to 1% graphite. For both powders, the same trend is observed: the faster the carbon starts to diffuse, the lower the growth caused by the copper melting. It can also be observed that the higher the temperature required to initiate carbon diffusion, the larger the effect of a slight shift of that temperature on the copper melting growth. This can be explained by the fact that the later the carbon starts to diffuse, less time the carbon has to diffuse totally in the iron lattice prior to copper melting. The level of carbon in the austenite is likely inversely proportional to the temperature at which the carbon starts to diffuse. The growth caused by copper melting is strongly affected by the level of carbon in the steel matrix. Therefore, a powder mix for which diffusion of carbon is relatively slow will show much higher sensitivity to sintering conditions. This illustrates that the selection of an adequate graphite grade is very important to achieve better dimensional control, especially if difficult diffusion conditions exist (powder selection, sintering conditions, temperature profile, atmosphere, dew point, etc...).

III. Effect of heating rate.

Results discussed in the previous sections clearly showed the importance of the transport phenomena on the dimensional behaviour of specimens, in particular the carbon diffusion. The temperature and time are key factors when diffusion is involved. Therefore, tests were carried out at a higher heating rate to evaluate the influence of less time during heating on the dimensional behaviour of FL-4605 and FLC-4608 mixes. Figure 13 shows the dilatometric

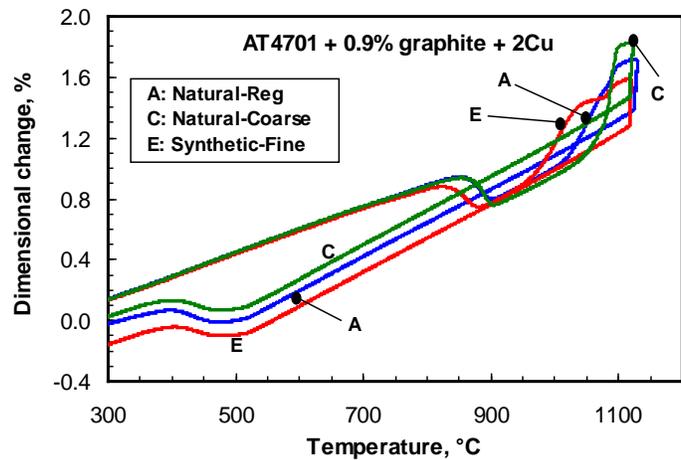


Figure 11. Dilatometric profiles of specimens of ATOMET 4701 with 2%Cu and different grades of graphite.

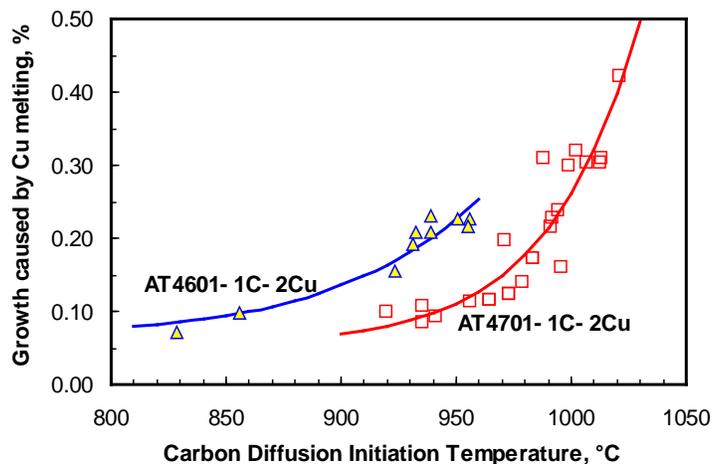


Figure 12. Relation between the growth caused by Cu melting and the temperature at which carbon starts to diffuse in the austenite.

profiles of ATOMET 4601 with 0.6% graphite (synthetic grade E) obtained at a heating rate of 10 and 20°C/min. Increasing the heating rate from 10 to 20°C/min slightly delayed the $\alpha \rightarrow \gamma$ transformation by about 15°C. However, no significant effect is seen on the carbon diffusion that is effective right after the $\alpha \rightarrow \gamma$ transformation. A major difference is seen when carbon diffusion is completed, at temperature above 980°C. Indeed, the expansion rate is more important for the specimen heated at 20°C/min. This is explained by two competitive phenomena that take place above 980°C. The first phenomenon is the linear expansion rate, an intrinsic property of a material that is affected by the temperature, not by the time and thus, the heating rate. For austenite, the linear expansion rate is about 0.0022-0.0024%/°C. The second phenomenon that causes the shrinkage when pores are present in the part is the reorganization and movement of atoms in the lattice. The diffusion rate of atoms is given by a displacement divided by time and is thus, time sensitive. Less time results in less atoms movement and thus, less shrinkage. It should be noted that the diffusion rate of atoms increases with an increase in temperature. For FL-4605 specimens heated at 10 and 20°C/min, the rate of shrinkage due to atom diffusion was respectively 0.010 and 0.020%/min between 1000 and 1100°C. Finally, it is seen that changing the heating rate contributes to modify the dimensional change of specimens before and after maintenance at the maximum temperature. This shows that heating rate may affect the final size of a part

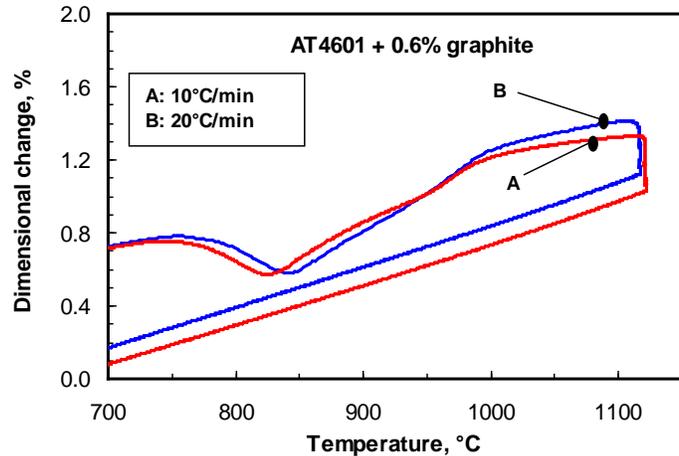


Figure 13. Effect of heating rate on the dilatometric profiles of FL-4605 specimens.

Figure 14 shows the dilatometric profiles of specimens of ATOMET 4601 containing 2% copper and 1% graphite. Natural graphite was used instead of synthetic graphite for these specimens. As was the case with the FL-4605 specimens with synthetic graphite, an increase in heating rate delayed the $\alpha \rightarrow \gamma$ transformation, the shift being slightly more pronounced with natural graphite, ~30°C. The diffusion of carbon in the γ phase also started at a higher temperature, 950°C versus 925°C, which was not really the case with the synthetic graphite. Natural graphite is therefore slightly more sensitive to heating rate. The growth associated with the carbon diffusion was more pronounced at 20°C/min. Chemical analysis showed that both specimens contained the same amount of carbon after the test, ~0.85%. The higher growth is therefore not related to the diffusion of higher amounts of carbon in the iron lattice. The lower amount of shrinkage associated with the diffusion of iron atoms due to a reduction in time can partly explain the increase in growth. However, it is estimated from figure 13 that the difference in shrinkage due to heating rate should be of the order of 0.03%, which corresponds only to 1/3 of the difference in growth caused by carbon diffusion between 10 and 20°C/min. Heating rate seems therefore to affect how the austenite structure dissolved the carbon and expanded.

Increasing the heating rate also significantly reduced the growth associated with the Cu melting. This is contrary to the trend observed and discussed in the previous section where an increase of the starting temperature for carbon diffusion leads to an increase of the copper melting growth. As mentioned earlier, the level of sintered carbon was the same for both specimens. It appears therefore that heating rate affects the way copper penetrates into the iron particles and in particular between the steel particles.

As was the case with FL-4605 specimen, the dimensional change of the specimen heated at 20°C/min was slightly more positive than those heated at 10°C/min before starting the sintering at 1120°C. However, it is seen that after sintering at 1120°C for 40 min, the dimension change is similar for both specimens. Figure 15 shows the variation of the dimensional change with time at isotherm. It should be noted that dimensional change is considered to be nil when sintering at isotherm starts, at 0 min. The dimensional change follows an exponential law as expected. It is seen that the shrinkage is more rapid in the specimen that was heated at 20°C/min. This can be explained by the fact that diffusion already took place to a much higher extent in the specimen heated at a lower rate. Because of the exponential nature of the diffusion process, it is therefore normal that the specimen heated at 10°C/min shrinks at a lower rate when sintering at isotherm starts. It should be noted that the shrinkage rate of the specimen heated at 20°C/min is greater between 0 and 15 min but is very similar above 15 min.

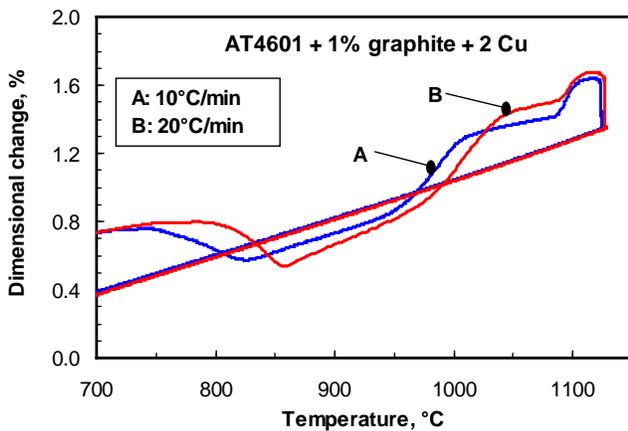


Figure 14. Effect of heating rate on the dilatometric profiles of FLC-4608 specimens.

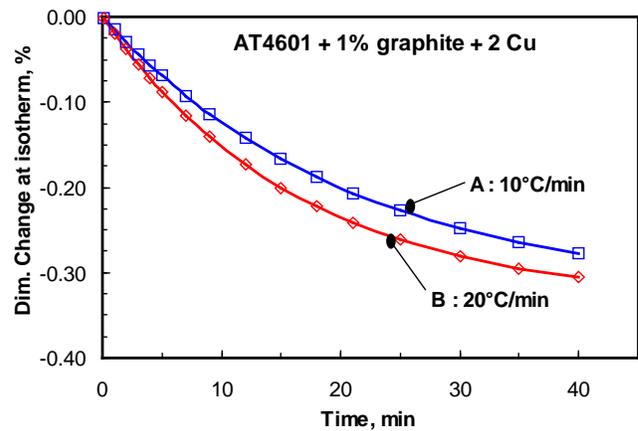


Figure 15. Effect of heating rate on the variation of the dimensional change with time at isotherm

d. Effect of lubricant type

All the results presented in the previous section were obtained on mixes containing waxes. However, it is known that the nature of lubricant can have a strong effect on dimensional change [6, 7]. Figure 16 shows the dimensional change versus green size for FL-4605 mixes containing different types of lubricant. It is seen that the dimensional change is slightly more positive when Zn stearate is used for both formulation, ~ +0.05%. However, using no lubricant has a much stronger effect on the dimensional change, which increases by +0.17% and +0.13% as compared to wax and Zn stearate respectively. Figure 17 shows the dilatometric profiles of specimens of FL-4605 containing synthetic graphite and wax, Zn stearate or no internal lubricant. It should be noted that specimens with lubricants were delubed prior to run the test and a heating rate of

20°C/min was applied to all the specimens. Significant differences are observed between the specimens. Using Zn stearate instead of wax as internal lubricant reduces the contraction associated with the $\alpha \rightarrow \gamma$ transformation and affects the carbon diffusion behaviour. Indeed, carbon diffuses at its maximum rate just after the completion of the $\alpha \rightarrow \gamma$ transformation and then tends to decelerate up to $\sim 1000^\circ\text{C}$. In the case of wax, carbon diffusion is maximized at much higher temperatures, between 950 and 1000°C . The carbon diffusion is completed at about the same temperature for both lubricants. Also, the dimensional change of specimens is very similar before reaching the maximum temperature. For the specimens without lubricant, the contraction associated with the $\alpha \rightarrow \gamma$ transformation is very small and the carbon diffuses easily in the γ lattice, as it was the case with Zn stearate. Most of the carbon diffuses between 875 and 920°C . As a result of these changes, it is seen that the dimensional change of the specimen without lubricant is significantly more positive as compared to that of specimens with lubricants. This difference is maintained at isotherm and during cooling down to room temperature.

Figure 18 shows the influence of the type of internal lubricant on the dimensional change variation with time at 1120°C . It is seen that the shrinkage is faster for wax as compared to Zn stearate. This difference explains the difference in dimensional change obtained after sintering, which is about 0.05%. The specimen without internal lubricant shows the lowest shrinkage rate at isotherm. It is known that a mix with no internal lubricant shows very poor

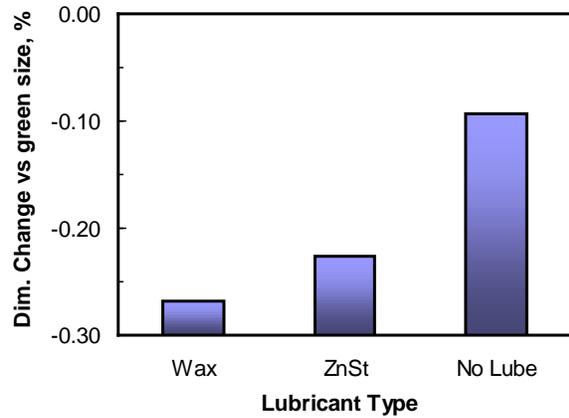


Figure 16. Effect of lubricant type on dimensional change after sintering 25 min at 1120°C .

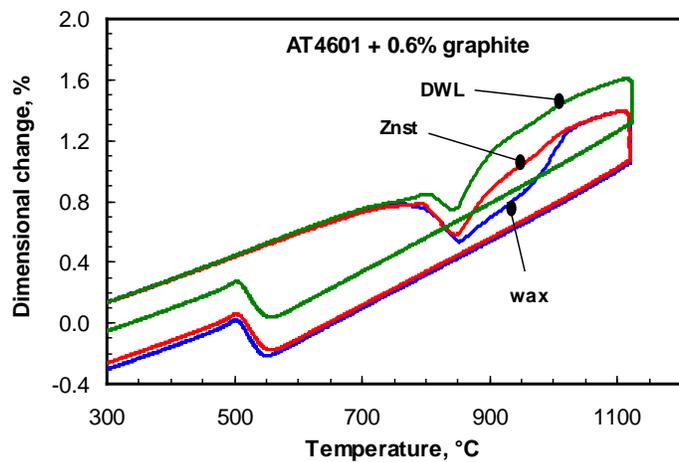


Figure 17. Effect of lubricant types on the dilatometric profiles of FL-4605 specimens.

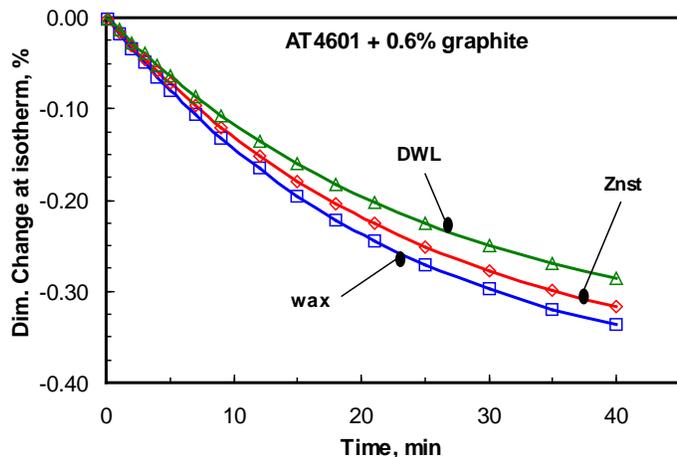


Figure 18. Effect of lubricant on the variation of dimensional change with time at isotherm for FL-4605 specimens.

particle rearrangement during pressing [16]. One hypothesis is that this poor particle rearrangement has contributed to reduce the particles to particles contacts, reducing therefore the iron diffusion process. Presence of internal lubricant helps increasing the inter-particles bonding, which contributes to accelerate the iron diffusion process, and thus the shrinkage. It appears clearly based on these results that the level of lubricant may have a strong effect on the dimensional behaviour of specimens. Nevertheless, this was not evaluated in this study.

CONCLUSIONS

The dimensional response during sintering of two sinter-hardening steel powders, ATOMET 4601 and 4701, admixed with various levels and types of graphite, copper and lubricant was investigated with a dilatometer at two different heating rates. The following conclusions can be drawn from this study:

- Dilatometry has proven to be a very powerful tool to better understand the phenomena occurring during sintering and helps to optimize the powder mix formulation.
- Adding graphite to a mix accelerates diffusion of iron atoms and shrinkage at elevated temperature.
- The nature and size distribution of graphite strongly affects the carbon diffusion and the dimensional change. In particular, synthetic graphite helps promote carbon diffusion in the iron lattice and a better inter-particle sintering.
- Size distribution has a strong effect on the carbon diffusion of natural graphite, while it has only a minute effect for synthetic graphite.
- Growth associated with the copper melting is strongly affected by the level of carbon in the iron matrix, the carbon diffusion rate and the level of inter-particle sintering. The copper melting growth is minimised if carbon diffusion in the austenite takes place at low temperatures, typically below 900°C.
- The final dimension of a part is directly related to the growth caused by copper melting. The selection of an adequate graphite grade is critical in order to minimize the variation in dimensional change caused by the copper melting.
- The type of lubricant affects the $\alpha \rightarrow \gamma$ transformation, carbon diffusion and the shrinkage at isotherm. This explains the differences in dimensional change obtained during normal sintering process with different lubricants.
- Using no internal lubricant strongly affects the $\alpha \rightarrow \gamma$ transformation and helps promote carbon diffusion. On the other hand, it slightly impedes shrinkage at the maximum sintering temperature.

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