Fixed and Variable Temperature Super-Solidus Liquid Phase Sintering of High Chromium Cast Iron with 25 Wt.%CR and Its Microstructure

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Abstract: A variable temperature super-solidus liquid phase sintering (SLPS) technique is employed in fabrication of high chromium cast iron (HCCI) with 25 wt.%Cr to extend its sintering temperature window. Its microstructure evolution, mechanical properties, and abrasive wear behavior are investigated systemically. The results indicate that the variable temperature SLPS can obtain samples with full density plus fine and uniformly distributed carbide particles, and its carbide volume fraction is increased by 4~5% in comparison with the fixed temperature SLPSed one. Meanwhile, its bend strength and impact toughness can be raised by 8.0% and 16.7%, respectively. Finally, the sintering temperature window for variable temperature SLPS of HCCI is extended by 12 °C, reaching 27 °C.

Keywords: high chromium iron; SLPS; microstructure; mechanical property; impact abrasive wear

1. Introduction

As a third-generation wear-resistant cast iron, high chromium cast iron (HCCI) has an outstanding performance with the combination of the hard M7C3 carbides and the tough matrix [1–3]. The volume fraction of carbides, their type, morphology, orientation, and the properties of the matrix are the vital factors to control the wear resistance of HCCI [3–8]. As such, the wear resistance of the alloy can be significantly improved by adding alloying elements (like W, B, Mo, Ti), but the numerous inevitable chrysanthemum-like carbides with sharp edges in normal cast HCCI always become the pores and stress concentration raisers, which deteriorates its toughness [9,10]. Presently, a hypoeutectic high chromium cast iron is prepared by P/M process with pre-alloyed powders [11]. The results indicate that samples with full density could be fabricated, the sintering temperature window suitable for SLPS is around 15 °C, and the microstructure of sintered hypoeutectic HCCI is of square bars of M7C3 carbides that are finely distributed in a mainly martensitic microstructure. It significantly changes the morphology of carbides and effectively reduces their consecutiveness in space, and hence effectively increases HCCI strength and toughness. The bending strength (2241 MPa) and impact toughness (8.0 J/cm²) of sintered HCCI in optimized sintering parameters are over twice that of normal HCCI (bending strength of 900 MPa, impact toughness of 4.0 J/cm²). Meanwhile, the hardness of sintered HCCI is not inferior to or even higher than that of normal HCCI. The results also show that as sintering temperature or holding time is raised, both grain and carbides are gradually coarsened. Of course, there is a huge impact of the sintering temperature and holding time on the alloy’s mechanical properties. Apparently, only strictly controlling the sintering temperature and holding time can prepare a full-density product with a good combination of strength and toughness.

Generally, SLPS is especially suitable for enhanced densification of alloys with pre-alloyed powder as raw materials, and the sintering window for SLPS should be within a temperature interval where solid and liquid phases coexist. In the case of SLPS of
HCCI, raising sintering temperature could increase the volume fraction of liquid phase for promoting the particle rearrangement and liquid phase penetration into the pores between particles to facilitate rapid densification, but it also comes with the risk of coarsened grains and compact deformation. Conversely, lowering sintering temperature could make the carbides’ precipitation more sufficient and reduce the solubility of alloying elements in the matrix, which would enhance the martensite transformation starting temperature (Ms) and, as a result, the matrix is dominated by more martensite. However, too low a sintering temperature would reduce the volume fraction of liquid phase in the SLPS process, leading to products with poor density. Therefore, a wide available sintering temperature window is the guarantee of stable reproduction by SLPS, but it usually is around 10–15 °C, just as found in our previous paper [11]. Hence, how to extend the temperature window has become one of the hot research areas in SLPS. Adding some trace elements has been reported to be an effective method to expand the window [12,13]. However, it is not easy work to find a feasible trace element that might not cause increased cost also. Because the densification process of SLPS could be adjusted by variable sintering temperature, a variable temperature sintering for SLPS of HCCI is proposed by us. Compared with the conventional fixed temperature sintering process mentioned in our previous paper [11], variable temperature SLPS consists of two-temperature sintering, that is, high temperature sintering for a short time and low temperature sintering for a long time. High temperature sintering for a short time has two advantages: one is to enhance densification of a compact resulting from a high-volume fraction of liquid phase, and the other is to keep it from developing coarsened grains plus deformation because of a short holding time. Low temperature sintering for a long time could ensure full precipitation of carbides, which would lead to a matrix with a higher Ms and more martensite arising from its lower solubility of alloying elements. In theory, this new sintering technique not only extends the temperature window but also fabricates HCCI components with high precision, full density, high hardness, and optimized microstructure.

In this work, a hypereutectic HCCI was melted and then water atomized into powders as raw materials. Its compacts were densified by SLPS of fixed temperature and variable temperature, respectively. Then, a comparison between the samples fabricated by the two different SLPS methods was carried out systematically, including densification, microstructure evolution, mechanical property, and abrasive wear resistance.

2. Materials and Methods
2.1. Experimental Details

A hypereutectic HCCI was prepared by pressing plus super-solidus liquid phase sintering (SLPS) water atomized pre-alloyed powders, and its final chemical composition, as shown in Table 1, was measured by an optical emission spark spectroscopy. Prior to densification, the particles size was analyzed by a BT-9300H Laser (Baite Instrument Co., Ltd, Dandong, China) particle size analyzer and the size distribution range (D_{10}, D_{50}, and D_{90}) was listed in Table 2. One percent styrene-butadiene rubber (SBR) was mixed with the raw powders as lubricant to improve their compacting ability. Green compacts were first pressed with a pressing pressure around 300 MPa on a SFLS manual hydraulic press, and then SLPS technique was adopted to sinter specimens in a GSL1600X vacuum tube furnace (Kejing Material Technology Co., Ltd, Hefei, China) under a vacuum (0.01 MPa) with a temperature accuracy of ±5 °C.

Table 1. The chemical composition of the HCCI (wt.%).

| Elements | C   | Cr  | Mn  | Si  | Fe    |
|----------|-----|-----|-----|-----|-------|
| wt.%     | 3.47| 26.40| 0.50| 1.20| balance |
2.2. SLPS of the HCCI

Differential thermal analysis (DTA) of the water atomized HCCI powders was performed in an alumina crucible of a Sta449C simultaneous differential thermal analyzer (NETZSCH Scientific Instruments Trading Ltd., Shanghai, China) at a rate of 10 K min\(^{-1}\) in an argon atmosphere. The results indicated that the alloy’s temperature range of eutectic transformation was 1148.0–1275.8 °C. Hence, SLPS of the green compacts would be performed in this temperature interval by variable sintering temperature, while holding time was kept the same, 90 min. The temperature-time profile of fixed temperature SLPS was as shown in Figure 1a with variable sintering temperature. Finally, an optimized sintering temperature window (OSTW) would be obtained on the result of the density, microstructure, and mechanical properties of the sintered samples. The temperature-time profile of variable temperature SLPS was also as shown in Figure 1a, which contained ceiling temperature sintering for 15 min and floor temperature sintering for 65 min. Here, ceiling and floor temperatures corresponded to the upper and lower limits of OSTW in fixed temperature SLPS and were plus or minus 5–10 °C, respectively. The total holding time in variable temperature SLPS was the same in the fixed temperature one, as shown in Figure 1b.

![Figure 1](image_url)

**Figure 1.** Schematic of the SLPS of two sintered HCCI: (a) whole diagram, (b) partial enlargement.

As shown in Figure 1a, the tube furnace was first heated to 250 °C and 450 °C for 30 min each time for compacts to degas and debind, respectively. Then holding at 1000 °C for 30 min was conducted to get an even temperature field in them. After that, the heating or cooling rate was 2 °C min\(^{-1}\) until it reached final sintering temperature. During variable temperature SLPS, the cooling rate from ceiling to floor temperature was also 2 °C min\(^{-1}\). The total soaking time for the two kinds of SLPS was the same: 90 min. After sintering, it was cooled to 800 °C at a rate of 8 °C min\(^{-1}\) and then went through furnace cooling to room temperature.

The ceiling and floor temperatures were set on the ground of the OSTW of fixed temperature SLPS, and the final designed parameters of variable temperature SLPS for experiment were as listed in Table 3. The total time from the onset of ceiling temperature sintering to the end of the floor temperature one was kept at 90 min by adjusting the cooling rate of the temperature interval between them.

| Table 2. The size distribution of the raw powders. |
|-----------------------------------------------|
| Water atomized HCCI powder | D\(_{10}\) (μm) | D\(_{50}\) (μm) | D\(_{90}\) (μm) |
|-------------------------------|----------------|----------------|----------------|
|                               | 13.24          | 36.25          | 85.90          |

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Table 3. The designed sintering parameters for variable temperature SLPS.

| Specimens | #0      | #1      | #2      | #3      | #4      | #5      | #6      |
|-----------|---------|---------|---------|---------|---------|---------|---------|
| Ceiling   | 1273 °C | 1270 °C | 1270 °C | 1267 °C | 1267 °C | 1267 °C | 1267 °C |
| Floor     | 1240 °C | 1240 °C | 1240 °C | 1240 °C | 1240 °C | 1245 °C | 1235 °C |
| temperature | × 15 min | × 20 min | × 15 min | × 20 min | × 15 min | × 15 min | × 15 min |
| Floor     | 1240 °C | 1240 °C | 1240 °C | 1240 °C | 1240 °C | 1245 °C | 1235 °C |
| temperature | × 65 min | × 65 min | × 65 min | × 65 min | × 65 min | × 65 min | × 65 min |

The density of the sintered HCCI samples was measured by Archimedes method (drainage volume method) and their microstructure was analyzed using a Leitz-MM6 optical microscope (OM) and/or a FEI QUANTA200 environmental scanning electron microscope (SEM). The volume fraction and roundness of carbides in the HCCI were measured quantitatively with analysis software of Image-Pro Plus and their final results were the statistical average values of at least more than 6 microstructure images with 200 magnification [14]. The smaller the value of roundness, the more the target pattern tends to be round. The theoretical minimum value of roundness is 1, as shown in Figure 2.

The roundness reflected the spheroidization of carbides and was described as follows:

$$C(\text{roundness}) = \frac{P^2}{4 \times \pi \times A} \quad (1)$$

where P was the mean perimeter of carbides, A was the mean area of carbides.

Figure 2. Schematic of the roundness of carbides.

The phase constitution of the alloy was analyzed by a D8-advance (Brooke Technology Co., Ltd, Beijing, China) X-ray diffraction instrument (XRD, Cu target, λ = 0.15405 nm) at a scan rate of 4°/min within the angle range from 30° to 100°. Mechanical properties of the sintered HCCI covered hardness, impact toughness, and bending strength. The hardness measurement was conducted by a DUR-O-Test and each result was the average of more than 5 inspections. Bending strength and impact toughness were tested on an Instron3369 universal mechanical testing machine (ITW group Instron company, Norwood, MA, USA) and an MLD-10 dynamic load abrasion impact tester, and its abrasion wear resistance was reported in terms of volume loss, mm³. More information about the dynamic load abrasion impact tester is available in our previous paper [11].

$$\text{Volume loss (mm}^3\text{)} = \frac{Q}{D} \times 1000 \quad (2)$$

where Q was the total mass loss (g) of a specimen after impact abrasive wear test for 60 min, D was its density (g/cm³).
The size of specimen for mechanical property evaluation was (a) 5 mm × 5 mm × 35 mm for bending strength; (b) 5 mm × 5 mm × 50 mm for impact toughness; and (c) 10 mm × 10 mm × 30 mm for impact abrasive wear tests. They were first wire-electrode cut and then surface grinded into the required size.

3. Results and Discussion

3.1. Effect of Sintering Temperature on Microstructure Evolution

Firstly, fixed temperature sintering is carried out to evaluate systematically the influence of sintering temperature on densification, microstructure evolution, and mechanical properties of the alloy. The relationship between the density and hardness of the sintered specimens and sintering temperature is presented in Figure 3 (holding time of 90 min). It reveals that the changes of both density and hardness perform almost in the same way as a function of sintering temperature. It rapidly rises at first and then reaches a stable peak when the sintering temperature is higher than 1250 °C, as shown in Figure 3a, and finally gradually declines if the sintering temperature is further increased. Relative density could be of more than 98.5% in comparison with 7.6 g/cm³ (the density of as-cast HCCI with the same chemical composition). Raising the temperature would increase the liquid phase's volume fraction in SLPS and enhances rearrangement of solid particles and infiltration of liquid melt into the pores between them in a green compact, which is beneficial to densification of specimens. By the way, it is also discovered that deformation of sintered components begins to take place at temperatures over 1265 °C due to the formation of excessive liquid melt.

The microstructure evolution of the alloy with sintering temperature is represented in Figure 4. It consists of white carbide particles and grey matrix grains. The white particles are precipitated mainly along the grain boundary of the matrix, in addition to some amount of small ones inside grains, and become coarsened obviously as the temperature increases.
Meanwhile, their morphology is changed from rods plus some small granules to distinctly coarsened massive bulks with small aspect ratio and some sharp edges, which should be the result of Oswald ripening during SLPS and deleterious to the alloy’s strength and toughness [15]. The influence of sintering temperature on volume fraction of carbides in the alloy is shown in Figure 3c, which indicates that it gradually increases as sintering temperature is lowered in the interval.

![Figure 4](image-url)

Figure 4. The microstructure evolution of fixed temperature SLPSed HCCI at (a) 1250 °C, (b) 1255 °C, (c) 1260 °C, (d) 1265 °C.

As shown in Figure 3a, specimens sintered at 1245–1265 °C are of a stable and high density and hardness with little deformation—around 7.53–7.54 g/cm³ and HRC 60–62.5, respectively. Hence, they are chosen to conduct further mechanical property evaluations and their bending strength and impact toughness are given in Figure 3b. The results in Figure 3b imply that both bending strength and impact toughness signal decrease when sintering temperature is altered by not more than 20 °C, which means that there is a huge impact of sintering temperature on the alloy’s mechanical properties. The reason why SLPS sintering temperature put such a strong influence on the properties could be due to carbide coarsening and its morphology changes just as discussed above. The formation of coarsened bulks with sharp edges would intensify stress concentration and deteriorate the alloy’s strength and toughness.

Based on the results of fixed temperature sintering and discussions presented above, one conclusion that could be reached is that the sintering temperature window available for the fabrication of the HCCI by SLPS is around 10 °C from 1250–1260 °C and the optimized sintering temperature (OST) of fixed temperature sintering is around 1250 °C (mark A). Such a narrow window would pose a challenge for the alloy’s industry production due to accurate temperature control.

Variable temperature SLPS is then conducted for the purpose of broadening the sintering temperature window available and improve the carbides’ morphology. The density, hardness, carbide volume fraction, and roundness of variable temperature SLPSed HCCI samples are reported in Table 4. Corresponding parameters of the one fabricated at optimized temperature by fixed temperature SLPS is also listed in it for comparison.
Table 4. The statistical results of OM images.

| Specimens | Volume Fraction of Carbides (Vol.%) | Relative Density (%) | Roundness of Carbide |
|-----------|-------------------------------------|----------------------|----------------------|
| A         | 35.3                                | 98.6                 | 2.76                 |
| #1        | 39.6                                | 98.4                 | 4.04                 |
| #2        | 39.9                                | 98.2                 | 3.20                 |
| #3        | 39.9                                | 98.6                 | 4.03                 |
| #4        | 39.8                                | 98.6                 | 2.61                 |
| #5        | 40.8                                | 98.4                 | 3.25                 |
| #6        | 40.9                                | 98.5                 | 3.18                 |

Relative density around 98.5% can be certainly reached by this new SLPS technique and no sample deformation due to high temperature sintering is found when ceiling temperature is below 1270 °C. An important conclusion can be drawn from the results in Table 4: that the sintering temperature window available for this technique to fabricate samples with full density can be extended to 40 °C. Ceiling temperature sintering for 15 min ensures effective densification of a compact. In addition, the floor temperature sintering for 65 min can obtain HCCI with higher volume fraction of finer carbides (lower roundness).

The microstructures evolution of variable temperature SLPSed HCCIs with different sintering parameters is presented in Figure 5. It still consists of white carbide particles and grey matrix grains, and the characters of carbides, such as distribution and morphology, is similar to that in the fixed temperature SLPSed one. Nevertheless, the results in Figure 5 indicate that full precipitation of carbides resulted because of the floor temperature sintering, and the carbide volume fraction increased by 4.3–5.6% in comparison with that of fixed temperature SLPSed alloy. SLPS of the HCCI is implemented in the temperature interval of its eutectic transformation. So, reducing sintering temperature is beneficial to eutectic transformation accomplishment and there would be more carbides in the alloy.

In addition, the average roundness of carbide particles in Figure 5 (3.38) is smaller than that in Figure 4 (3.62), which means that there are more fine white carbide granules left in the matrix grains of variable temperature sintered HCCIs. Carbide particles’ coarsening
during SLPS is mainly through Oswald ripening because they are immersed in melt liquid. In theory, the higher the sintering temperature, the more serious the coarsening. However, such a law could little be found in Figure 5 because there is only a very short time ceiling temperature sintering in variable temperature sintering. In fact, its floor temperature is lower than the sintering temperature of fixed temperature SLPS. Hence, Oswald ripening of carbide granules in variable temperature sintered HCCI is obviously less serious than that in the fixed temperature sintered one.

3.2. Mechanical Property

Hardness, bending strength, and impact toughness of variable temperature SLPSed samples, as well as the one fixed temperature SLPSed at OST, are presented in Table 5. It is quite interesting that the hardness of all samples listed in Table 5 is almost the same, although there is an apparent carbide volume fraction difference between samples SLPSed by the two techniques. The matrix of sintered HCCI is mainly of martensite [16,17]. The reason for the stable hardness phenomenon is because of the trade-off between carbide volume fraction and carbon supersaturation in martensite. That is, there is always a martensite matrix with low carbon supersaturation and hardness in a sintered HCCI with high carbide volume fraction. Phase diagram calculation (Thermo-Calc V5.0, TCFE 6 database) analysis for the alloy is also performed by the authors, as shown in Figure 6. The composition of the metal specified in the simulation is the same as Table 1. It reveals that a decrease in the solubility of these elements leads to an increase in the proportion of carbides as temperature drops gradually during solidification.

Table 5. The mechanical properties of investigated specimens.

| Specimens | Hardness (HRC) | Bending Strength (MPa) | Impact Toughness (J/cm²) |
|-----------|----------------|-----------------------|-------------------------|
| A         | 62.2           | 1755.6                | 4.2                     |
| #1        | 62.6           | 1899.2                | 3.2                     |
| #2        | 62.4           | 1776.9                | 4.0                     |
| #3        | 62.7           | 1816.8                | 3.5                     |
| #4        | 62.1           | 1895.9                | 4.9                     |
| #5        | 62.0           | 1792.7                | 4.0                     |
| #6        | 62.3           | 1569.5                | 4.1                     |

Nevertheless, the results in Table 5 clearly indicate that both bending strength and impact toughness are sensitive to the volume fraction and morphology change of carbides in the alloy. Their highest value set is of 1895.9 MPa and 4.9 J/cm² in sample #4, which represents an increase of 8.0% and 16.7%, respectively, in comparison with sample A.
Meanwhile, the variation range of bending strength and impact toughness of variable temperature SLPSed samples are 1569.5–1899.2 MPa and 3.2–4.9 J/cm², severally, corresponding to 1381.7–1755.6 MPa and 2.1–4.2 J/cm² of the fixed temperature SLPSed ones reported in Figure 3. Apparently, the lower roundness of carbide could effectively weaken stress concentration and extend crack propagation path length, which is beneficial to the improvement of impact toughness. Therefore, variable temperature SLPS could provide HCCI product with better and more stable performance.

It could be also revealed by comparing carbide granules in both Figures 4 and 5 that the average size of carbides in the latter is not only smaller, but there are little sharp edges in their morphology. Furthermore, there are many fine white particles in matrix grains in Figure 5, which reconfirms that Oswald ripening of carbide granules in variable temperature sintered HCCI is less than that in the fixed temperature sintered one. SLPS belongs to liquid phase sintering. Hence, lowering sintering temperature, such as in floor temperature sintering, could reduce the amount of liquid melt in the sintering process and retard both carbide and grain coarsening [18,19]. Carbide particles with round edges could effectively weaken stress concentration in service and extend crack propagation path length, leading to improvement of both strength and toughness of the sintered alloy. However, the secondary carbides have little effect on the mechanical properties since the specimens we investigated had not suffered heat treatment.

The results of Figure 5 and Table 5 indicate that the sintering temperature window available for variable temperature SLPS of the HCCI alloy is around 27 °C from 1240–1267 °C, which means that its sintering temperature window is effectively broadened by 17 °C. Therefore, this new technique would surely benefit stable production of high quality sintered HCCI products in industry.

The XRD results of raw powders and sintered HCCIs is shown in Figure 7. It reveals that there are mainly austenite and M₇C₃ carbide, as well as a small amount of martensite, in the raw materials, but there is chiefly martensite in the matrix of sintered alloys, which reconfirms our report in a previous paper [11].
To further probe the difference of fracture failure mechanism between the sintered HCCIs manufactured by the two different SLPS methods, the morphologies of their fracture planes are examined by SEM and EDS as exhibited in Figures 8 and 9, and Table 6. The fractograph of specimen A in Figure 8a,b demonstrates a multitude of straight cleavage facets triggered by localized deflection of cleavage cracks along the duplex boundary, in which, mainly, are the primary carbides-martensite matrix interfaces. However, in specimen #4 (in Figure 8c,d), there are not only intergranular fractures across the M7C3 network structure but also a few transcryalline ones in the carbides, which is confirmed by EDS, as shown in Figure 9 and Table 6. It is worthy to point out that a few micro-sized dimples resulted from the cracks propagating along the interfaces between a fine carbide and a martensite matrix appearing in Figure 8d. Plenty of straight cleavage facets could be found in Figure 8b. Meanwhile, there appear many lacerated ridges at cleavage facets in Figure 8d, which illustrates that more energy would be required for cracks in specimen #4 to propagate. Therefore, its impact toughness (4.9 J/cm²) is superior to specimen A’s (4.2 J/cm²).

Figure 8. Fractograph of (a,b) specimen A, (c,d) specimen #4.

Table 6. Statistical results of elements of EDS tests.

| Element | Wt.% | At% |
|---------|------|-----|
| FeK     | 32.53| 21.44|
| CrK     | 51.98| 36.80|
| CK      | 13.28| 40.70|
| MoL     | 1.98 | 0.76 |
4. Conclusions

In the presented work, P/M HCCI is fabricated by both fixed temperature and variable temperature SLPS methods. Their impact on microstructure evolution, mechanical properties, and wear behavior, as well as the temperature window available for sintering, are investigated and compared with each other, respectively or systematically. On the basis of the results, the following conclusions can be arrived at:

1. Variable temperature SLPS is a practical technique to manufacture HCCI with full density and could effectively widen the temperature window available in the alloy’s SLPS.
2. Compared with the fixed temperature SLPSed HCCI, the variable temperature sintered one is of higher carbide volume fracture and has more uniform distribution of finer carbide particles in the microstructure. Hence, it can perform better in both bending strength and impact toughness.

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