



Effect of Carbon on the Melting Behavior of a Mold Powder under High Heating Rates

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Abstract: Mold powders used for the continuous casting of steels contain different carbon carriers controlling the melting behavior. During melting, carbon particles may accumulate at the liquid slag pool surface resulting in steel recarburization, which significantly changes the product quality. Thus, to reduce or prevent this reaction, mold powders with considerably reduced carbon content, or even without free carbon, are required. To replace carbon with another melting-controlling component, the effect on the melting behavior of mold powders under near-process conditions, such as high heating rates, should be investigated. Consequently, a different procedure was developed to evaluate the effect of various carbon contents on the melting behavior. In this contribution, a granulated ultra-low carbon (ULC) mold powder was selected, and different graphite contents (0, 1, 2, and 5%) were added. Furthermore, a sample obtained by granule disintegration and 5% graphite addition was produced and compared with a powdery sample. Subsequently, the steel crucibles were covered to reduce oxygen supply, filled with the samples, inserted into a preheated furnace (700–1300 °C), held at the selected temperatures for 10 min, and quenched to room temperature. The samples were mineralogically investigated, and the experimental method was applicable for characterizing the melting behavior of mold powders. At lower temperatures, carbon reduces the reactions between raw material components owing to reduced particle contact and prevents the formation of new solid (for example, cuspidine) and liquid phases. With increasing carbon content, the reactions shifted to higher temperatures, further delaying liquid phase formation. This effect was more evident for the powdery mold powder than for the granulated mold powders. At elevated temperatures, the powdery sample contained a coherent liquid phase, whereas the granules

melted independently. This contribution provides a foundation for alternative melting-controlled additives for mold powders. It demonstrates that carbon substitutes should delay the reaction between raw material components and be stable until high temperatures.

Keywords: Mold powder, Free of elementary carbon, Melting behavior, Carbon replacement, SiC

Einfluss von Kohlenstoff auf das Aufschmelzverhalten eines Gießpulvers bei hohen Heizraten

Zusammenfassung: Gießpulver für den Strangguss von Stahl enthalten unterschiedliche Kohlenstoffträger in unterschiedlichen Mengen, die das Aufschmelzverhalten kontrollieren. Wird Kohlenstoff während des Aufschmelzens nicht vollständig oxidiert, reichern sich die Partikel auf der flüssigen Schlackenschicht an. In Kontakt mit der Stahlschmelze führen diese zu einem Aufkohlen des Stahles, was in weiterer Folge die Produktqualität negativ beeinflusst. Daher werden Gießpulver mit sehr niedrigem bzw. ohne freien Kohlenstoffgehalt benötigt. Um den Kohlenstoff durch eine geeignete Komponente zu ersetzen, muss zuerst dessen Auswirkung auf das Aufschmelzverhalten unter prozessnahen Bedingungen (z.B. hohe Heizraten) untersucht werden. Dafür wurde eine neuartige Versuchsdurchführung entwickelt: Um die Auswirkung unterschiedlicher Kohlenstoffgehalte auf das Aufschmelzverhalten zu untersuchen, wurden einem granuliertem ULC-Gießpulver unterschiedliche Mengen an Graphit zugesetzt (0%, 1%, 2%, 5%). Zusätzlich wurden die Granalien dieses Pulver zerstört und mit 5% Graphit vermischt, um den Effekt der Granulation zu untersuchen. Zur Reduktion der Sauerstoffzufuhr wurden die Proben in verschlossenen Stahltiegeln in den vorgeheizten Ofen (700–1300 °C) gestellt und anschließend mineralogisch untersucht. Die Ergebnisse zeigten, dass die Versuchsdurchführung für die Charakterisierung des Aufschmelzverhaltens von Gießpulvern geeignet ist. Bei niedrigen Temperaturen reduziert der zugesetzte Kohlenstoff einerseits die Bildung neuer fester Phasen (z.B. Cuspidin) durch Verminderung des Partikelkontakts, an-

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derserseits aber auch die Bildung einer flüssigen Phase. Mit zunehmendem Kohlenstoffgehalt werden die Reaktionen zu höheren Temperaturen verschoben. Dieser Effekt zeigt sich noch deutlicher bei der Pulverprobe. Während die Pulverproben bei hohen Temperaturen eine homogene Schmelzphase ausbilden, schmelzen Granalien unabhängig voneinander. Basierend auf diesen Ergebnissen muss der Ersatzrohstoff die Festkörperreaktion verlangsamen und bis zu hohen Temperaturen stabil sein.

Schlüsselwörter: Gießpulver, Kohlenstofffrei, Schmelzverhalten, Kohlenstoffersatz, SiC

1. Introduction

In the continuous casting of steel, different carbon carriers (e.g. graphite, carbon black) are used in varying quantities to control the melting behavior of mold powders. Depending on the steel quality, the carbon content of the mold powder is in the range of 2–20 wt%. Because of their non-wetting ability, carbon particles inhibit the formation of a continuous liquid phase by dispersing the liquid droplets (known as the skeleton effect). The melting behavior is dependent on the type and particle size of carbon used for mold powder fabrication [1–6].

Owing to the increasing temperature, reactions occur between raw material particles, forming new solid (including cuspidine and sodium calcium silicates) and liquid phases [6–10]. When carbon is not completely removed in the powdery and sintered layers (500–900 °C) during liquefaction by oxidation, it accumulates on the liquid slag pool surface in the mold. During casting, liquid steel may come into contact with carbon, resulting in steel recarburization and, consequently, reduced slab/bloom quality. Steel/carbon contact is caused by mold oscillation, steel flow from the submerged entry nozzle, and/or unsteady-state casting. To determine the effect of steel recarburization on its structure, the contact between molten steel, mold slag, and mold powder was evaluated [11], and the results corresponded with the observations from continuous casting. Therefore, to reduce the carbon content of the enriched layer, aggressive oxidation of carbon was performed using oxidizing agents such as MnO₂ and rapid burning-type carbons such as activated carbon. Alternatively, carbon was substituted with ceramic materials that are poorly wetted by molten slags [2, 11–16].

Thus, to minimize carbon uptake, mold powders with considerably reduced or without elemental carbon are required. Attempts to replace free carbon in mold powders with other melting-controlling additives have been reported [17–21], including nitrides (Si₃N₄, BN, TiN, MnN, Cr₂N, Fe₄N, AlN, and ZrN), carbides (SiC), and oxides (MgO, Mg₃Si₄O₁₀, Al₂O₃, SiO₂, CaO, TiO₂, Cr₂O₃, FeO, NiO, ZnO, and BaO). The effects of these oxides were investigated experimentally, and high sintering was observed in all cases [17]. However, nitrides were more effective, exhibiting a similar melting-controlling behavior to carbon. This was especially observed for BN, which has a similar crystal structure and physical and thermal properties to carbon.

Furthermore, its low wettability to slag and lower sintering compared with the other nitrides is advantageous. To control the melting rates, BN contents of 2–10 wt % and particle sizes of <0.3 mm were suggested. The addition of antioxidants further reduced the BN content. Subsequent industrial trials showed an increase in B of 2–3 ppm in the strand had a detrimental effect on product quality [17, 18]. Furthermore, the amount of free carbon was decreased to below 1 wt% in the mold powder using Si₃N₄ with a particle size of <0.5 μm and specific surface area of 2.2–3.5 m² g⁻¹. Steel recarburization was reduced to 4 ppm, without nitride reabsorption. The disadvantages of Si₃N₄ are its higher cost compared to carbon and relatively high gas formation during the melting process [19, 20].

Additionally, promising results were obtained by replacing free carbon with SiC. The experiments were performed in an argon atmosphere, and the pressed sample had an initial diameter and height of 4 mm, which was measured during heating. The reference materials were mold powder specimens with 2.0–2.5 wt% C. Their melting behavior was similar to that of powders containing 5.0–6.0 wt% SiC. Based on the melting curves (temperature vs. relative height), a suitable result for the continuous casting process is achieved when carbon is partially replaced by SiC. Samples were selected, and industrial trials were conducted, indicating that the typical three layers (the powder, sinter, and slag layers) differ from carbon containing mold powders and recarburization was reduced by 10% [22].

These investigations demonstrated the possibility of replacing carbon in mold powder. However, to date, the effect of its content has not been quantified at heating rates comparable to those of continuous casting. Furthermore, the reactions occurring at elevated temperatures should be elucidated. However, these results are necessary for selecting the most appropriate melting-controlling additives.

2. Experimental Procedure

Graphite with a grain size of maximum 2% >70 μm and d₅₀-value of 13 μm was used in this study. Samples were pre-

TABLE 1
Chemical composition of the ULC mold powder

Oxide	[wt%]
F	6.22
Na ₂ O	3.73
K ₂ O	0.10
CaO	36.17
MgO	5.00
Al ₂ O ₃	4.41
SiO ₂	37.12
P ₂ O ₅	0.38
SO ₃	0.37
Fe ₂ O ₃	2.60
BaO	0.07
C _{total}	3.78
C _{frei}	1.25

TABLE 2
Sample characterization

Sample	Description	Free carbon content (wt%)
MP1	As-received mold powder	1.25
MP2	As-received mold powder +1 wt% graphite	2.23
MP3	As-received mold powder +2 wt% graphite	3.19
MP4	As-received mold powder +5 wt% graphite	5.95
MP5	Granules dissolved in water +5 wt% graphite	5.95

pared based on a mold powder for the continuous casting of industrial ultra-low carbon (ULC) steel. The as-received mold powder was used as the reference sample (MP1), and its chemical composition is listed in Table 1. For MP2–MP4, different graphite contents (1–5 wt%) were added to the mold powder granules. Additionally, a powdery sample was prepared to determine the effect of granules during heating. The as-received mold powder granules were disintegrated by dissolving the binder component in water, followed by drying until a constant weight was achieved. Therefore, a powdery sample was obtained without changing the chemical composition or particle size of the raw material. Subsequently, graphite was added to the mixture (MP5). The investigated samples are listed in Table 2.

For thermal treatment, the furnace was heated to the selected temperature between 700–1300 °C, each specimen (~20 g) was weighed in a steel crucible (30 × 30 × 40 mm³), which was then closed with a lid and placed in the preheated furnace. After a dwell time of 10 min, the samples were removed from the furnace and quenched in air at room temperature. Subsequently, polished sections were prepared for mineralogical investigation using a reflected light microscopy and scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDX). The samples were ground for X-ray diffraction (XRD) analyses.

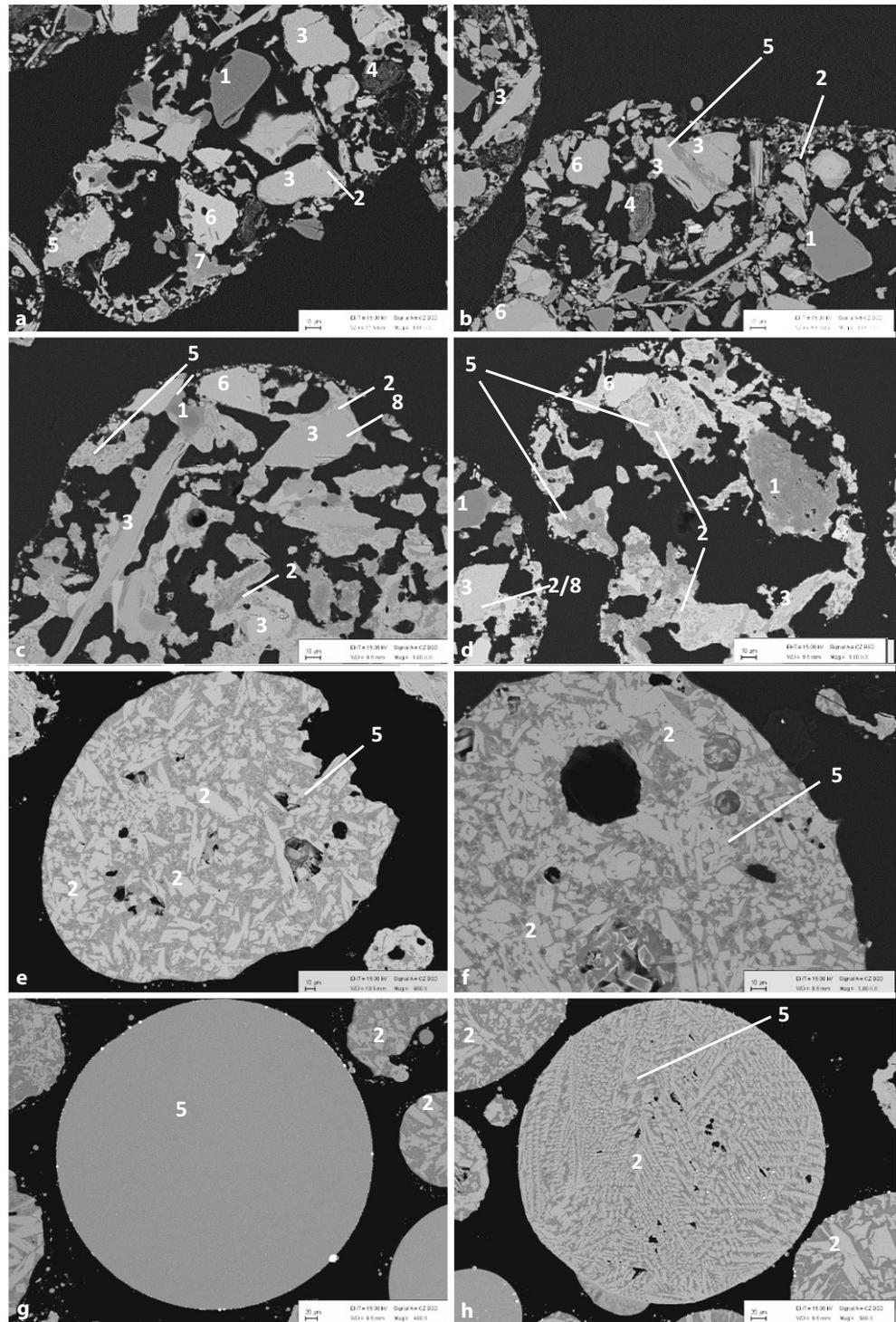
3. Results and Discussion

The main components of the original mold powder were wollastonite, albite, and fluorite, while smaller amounts of quartz, magnesite, calcite, hematite, diopside, and synthetic raw materials, such as phosphor and blast furnace slags, were detected. The minor phases were carbon black, cryolite, dolomite, and Ca₂SiO₄ (C₂S). After thermal treatment at 700 °C, the sample exhibited some changes in the initial composition. An increased CaO content was observed at the boundaries of the acidic components, such as quartz and albite. CaO originates from the onset of calcite decarburization at its surface, but calcite remains the main component of the particles. The decrease in the calcite decarburization temperature is attributed to SiO₂. Thus, the reaction occurred even at low temperatures. For the acidic components, an increase in Na₂O was also detected. Moreover, magnesite was decarburized. Furthermore, magnesium fluoride, which was observed up to 1000 °C, was formed owing to the diffusion of fluorine towards magnesia. In contrast, wollastonite, diopside, and phosphor slag did not undergo any reaction. At 900 °C

(Fig. 1a) the granule shape remained the same, but the particle edges became rounded, indicating the formation of a liquid phase. This is particularly true for components with a glassy phase. However, wollastonite, albite, and diopside were also involved, and cuspidine formed at their boundaries. For wollastonite, sodium calcium silicate was formed, separating the wollastonite in the particle center from the cuspidine edge. With increasing temperature, the contents of the fluorine-containing liquid phase and cuspidine increased, and at 1000 °C (Fig. 1c), only the residuals of larger particles were observed. Magnesium fluoride was dissolved in the liquid. At 1100 °C (Fig. 1e), the mold powder granules were liquified, and the only remaining solid phase was cuspidine. Large crystals were formed, indicating that they developed from solid phases. In contrast, small dendritic fluorite crystallized during the quenching process. Although the granules contained large amounts of the liquid phase, they remained separate from each other. This effect was also observed after thermal treatment at 1200 and 1300 °C. However, at 1200 °C (Fig. 1g), some granules had cuspidine crystals, whereas at 1300 °C, only the liquid phase was observed. This difference may be attributed to the higher fluorine and/or Na₂O content in the granules with only a liquid phase. Thus, the liquefaction temperature of the granules decreased. Similar melting behavior was observed for MP2.

Based on the reflected light microscopy images of MP3 and MP4, graphite was not completely oxidized in the annealed specimens. However, graphite oxidation increased with increasing temperature up to 1100 °C. At higher temperatures, the excess liquid phase and its rapid formation at the sample surface reduced the oxygen supply, resulting in reduced decarburization. At 700 °C, the dissociation of calcite and magnesite occurred. Additionally, the diffusion of Na₂O and CaO into the acidic components was observed, albeit to a lesser extent. Even at 900 °C (Fig. 1b), the granules had no distinct difference in appearance compared with those at lower temperatures. In contrast to the original mold powder, no liquid phase was formed, and the particle shape remained angular. The reaction boundaries of albite, quartz, and wollastonite appeared as narrow stripes at the particle edges. A considerable number of reactions were observed in the sample annealed at 1000 °C (Fig. 1d). A fluorine-containing liquid phase formed between the solid raw material particles, which accelerated cuspidine formation. Quartz was no longer stable, and a minimal amount of albite was detected. Residuals of large wollastonite, diopside, and fluorite particles were observed, and a few of these particles were detected at 1100 °C (Fig. 1f), where large cus-

Fig. 1: Back scattered electron images of MP1 and MP4 at different temperatures: MP1 at (a) 900 °C, (c) 1000 °C, (e) 1100 °C, and (g) 1200 °C and MP4 at (b) 900 °C, (d) 1000 °C, (f) 1100 °C, and (h) 1200 °C. 1—albite, 2—cuspidine, 3—wollastonite, 4—residual magnesite, 5—liquid phase, 6—fluorite, 7—quartz, and 8—sodium calcium silicate

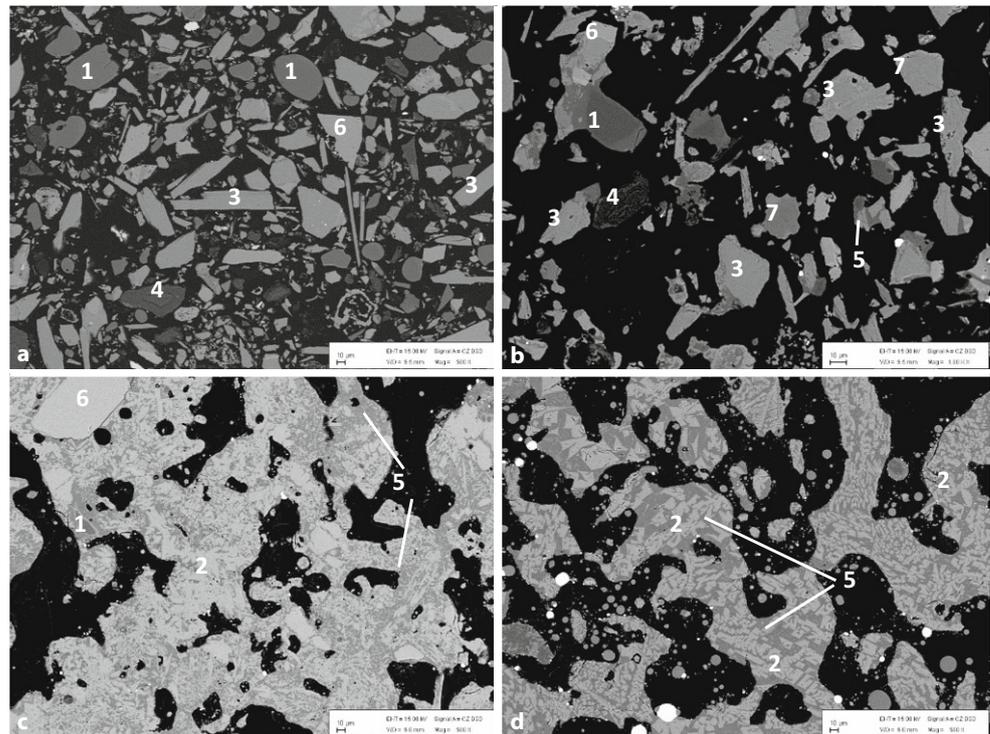


pidine crystals formed in the liquid phase. Additionally, the granules in this sample melted independently. After annealing at 1200 °C (Fig. 1h) and 1300 °C, graphite particles remained between the liquefied granules containing cuspidine as the only solid phase. A similar melting behavior was detected for MP3.

Therefore, increasing the carbon content delayed the reaction between the raw material particles. The formation

of the first liquid phase and new solid phases, for example cuspidine, shifted to higher temperatures. Although the graphite particles were large and did not directly contact the raw material particles, their effects were considerable owing to the presence of carbon black in the granules of the original mold powder. Thus, the reaction between the raw materials was prevented by the considerably smaller carbon black particles. Moreover, this clarifies the similar

Fig. 2: Back scattered electron images of MP5 at (a) 900 °C, (b) 1000 °C, (c) 1100 °C, and (d) 1200 °C; 1—albite, 2—cuspidine, 3—wollastonite, 4—residual magnesite, 5—liquid phase, 6—fluorite, 7—quartz



melting behavior of MP1 and MP2, as well as MP3 and MP4. Consequently, to increase the contact between the graphite and raw material particles, such as wollastonite, fluorite, and glassy phases, granule disintegration was required before carbon addition. Additionally, graphite was not completely oxidized after heat treatment, but differences in the melting behavior was observed. After annealing at 900 °C (Fig. 2a), the dissociation of calcite and magnesite was observed, and the latter indicates an increased fluorine content. The diffusion of CaO and Na₂O into the acidic components, and that of Na₂O and fluorine towards wollastonite, was also observed, as indicated by the narrow edge at the particle surface. The formation of a liquid phase at the interface of the phosphorus slag with other raw materials was observed after annealing at 1000 °C (Fig. 2b). Cuspidine was formed on the surfaces of the dissociated magnesite, albite, and wollastonite. A coherent liquid phase, which encapsulated the graphite particles, was observed at 1100 °C (Fig. 2c). Cuspidine formed large crystals and residual wollastonite, albite, and fluorite were partially retained. At 1200 °C (Fig. 2d), cuspidine was detected as the only solid phase, but it was liquefied at 1300 °C.

The analysis of the melting behavior of the granulated and powdery samples revealed that the effect of carbon addition at lower temperatures (< 1000 °C) is more evident in powdery samples. This may be owing to the increased contact between the raw material particles and carbon carriers in the powdery samples, which reduces diffusion and delays solid-solid reactions. In contrast, with increasing temperature, a continuous liquid is formed, independent of the carbon content. However, there were differences between the granulated and powdered samples. While the powdery

sample forms a coherent liquid phase, the granules melt independently, resulting in well-dispersed droplets.

Thus, the appropriate replacement of carbon in mold powders should fulfill the following requirements. At lower temperatures, the formation of new solid and liquid phases should be delayed by preventing diffusion. Additionally, carbon substitutes should be stable up to sufficiently high temperatures. Furthermore, poor wettability of the formed liquid phase is required to prevent the formation of a coherent liquid phase. Thus, nitrides and carbides are proposed for this purpose.

4. Summary

To quantify the effect of carbon on the melting behavior of mold powders, different graphite contents were added to a ULC mold powder that is used for the continuous casting of steel. The samples were annealed using high heating rates between 700–1300 °C and then quenched to room temperature. Subsequently, the samples were mineralogically investigated. With increasing carbon content, the solid-solid reactions between the raw material particles were delayed and shifted to higher temperatures. Despite their different graphite contents, MP1 and MP2, as well as MP3 and MP4, exhibited similar melting behaviors. Owing to the presence of graphite and carbon black in the mold powder granules, a reducing atmosphere was formed, and carbon black oxidation was delayed. Thus, the diffusion and formation of new solid or liquid phases was prevented at high temperatures. Furthermore, the addition of graphite to the granulated sample did not provide sufficient particle-graphite contact. Consequently, the granules

were disintegrated prior to the addition of graphite. The melting-controlling effect of carbon was more evident at temperatures <1000 °C, where solid-solid reactions were delayed owing to the increased contact between graphite and the raw material particles. Conversely, >1100 °C, the independent liquefaction of the granules prevented the formation of a coherent liquid phase, which is also the case for powdery samples at 1100 °C.

This contribution demonstrates the role of carbon in mold powders and provides a foundation for its replacement with other melting-controlling additives. Carbon substitutes should delay the reactions between the raw material components and be stable until high temperatures. Therefore, nitrides and carbides are proposed for this purpose in the future.

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