Effect of Firing Temperature on Mechanical Properties of $\text{Al}_2\text{O}_3\text{--C}$ Sensor for Continuous Measurement of Molten Steel Temperature

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$\text{Al}_2\text{O}_3\text{--C}$ sensor based on the blackbody cavity theory achieved a continuous measurement of molten steel temperature. Its mechanical properties were affected by the firing temperature. Firing temperature has great influences on the weight changing, decalescence/heat release reaction and structure evolution of the resin binder used for $\text{Al}_2\text{O}_3\text{--C}$ sensor. Results showed that the porosity increased and the strength declined below 750°C , main exothermic reaction and weight loss of the binder occurred. As the firing temperature increased to 800°C , a great enhancement of endothermic reaction and a drastic increase of crystallite size were observed, relating to the rapid shrinkage of the binder. The porosity decreased from 10.3% to 8.8% and the strength improved remarkably from 6.5 to 8.2 MPa in this temperature range. Strengths and porosities of the sensors fired under $800\text{--}950^\circ\text{C}$ were similar (8.2–8.5 MPa and 8.8%–8.4% respectively). Moreover, the temperature response and lifetime of the sensor fired at 800°C were almost equal to the sensor fired at 950°C according to industrial tests, which were about 165 s and 24 h.

KEY WORDS: firing temperature; $\text{Al}_2\text{O}_3\text{--C}$ sensor; molten steel temperature; mechanical properties.

1. Introduction

$\text{Al}_2\text{O}_3\text{--C}$ refractory is applied to a continuous temperature measurement sensor for molten steel because of its advantages such as excellent resistance to erosion, good thermal shock resistance, high thermal conductivity and low cost.^{1–4)} $\text{Al}_2\text{O}_3\text{--C}$ sensor achieved the continuous measurement of molten steel temperature, which is inserted into molten steel in a tundish, and then forms a blackbody cavity. It generates an infrared signal according to the different temperature that can be used to measure the temperature.³⁾ The compositions of the $\text{Al}_2\text{O}_3\text{--C}$ sensor are fused alumina powders, graphite flakes, phenolic resin and additives. Many investigators had done a lot of researches on preventing the carbon-containing refractory from oxidizing. On the one hand, Al, Mg, Si, B_4C , SiC and MgB_2 had been studied for the anti-oxidation additives.^{5–8)} On the other hand, the graphite flakes had been coated with MgAl_2O_4 , Al_2O_3 , SiO_2 , ZrO_2 and TiO_2 via sol-gel technique to improve the oxidation resistance and water-wettability.^{9–12)} Meanwhile, the manufacture processes are also of great importance on the performances of $\text{Al}_2\text{O}_3\text{--C}$ refractory as well as the compositions, which includes mixing, drying, cold isostatically pressed (CIP) formation, solidification, firing and mechanical fixing.

Firing has a vital influence on the weight loss, decalescence/heat release behavior and structure evolution of the resin binder in carbon-containing refractory. $\text{Al}_2\text{O}_3\text{--C}$ sensor ought to obtain a high strength, a low porosity and a rel-

atively stable binder through an appropriate firing temperature. The firing temperature is a key parameter of the firing. It is usually beyond 800°C , while the final temperature varies depending on the type of refractory.¹³⁾ The strength of refractory decreases rapidly with the changing of temperature in the range of 400 to 600°C when the phenolic resin is used as the binder.¹⁴⁾ The decreasing is caused by pyrolysis. The pyrolysis reaction converts the phenolic resin matrix to glassy carbon thus to create a rigid carbon skeleton. It contains complicated physicochemical processes, such as removing volatiles, formation of pores, shrinkage and structure evolution.^{15,16)} Actually, the mechanism of $\text{Al}_2\text{O}_3\text{--C}$ refractory firing is pyrolysis of the binder, while the rest compositions almost remain stable in firing. According to the firing temperature of the long nozzle, submerged nozzle and stopper rod, $\text{Al}_2\text{O}_3\text{--C}$ sensor is fired at 950°C in practice. However, there is no report and literature about the firing temperature of $\text{Al}_2\text{O}_3\text{--C}$ sensor. But the firing temperature affects the properties of the sensor, such as the strength, porosity, temperature response and service life. Indeed, $\text{Al}_2\text{O}_3\text{--C}$ sensor acquires poor properties when the firing temperature is not suitable. So it is necessary to study the appropriate firing temperature for $\text{Al}_2\text{O}_3\text{--C}$ sensor.

It is thus the aim of the present work to study the effect of firing temperature on mechanical properties and its mechanism of $\text{Al}_2\text{O}_3\text{--C}$ sensor, and then obtain a suitable firing temperature. This mechanism is investigated by evaluating the weight changing, decalescence/heat release reaction and structure evolution of the binder in firing process by differential scanning calorimetry (DSC), thermogravimetry-derivative thermogravimetry (TG-DTG) and X-ray diffrac-

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tion (XRD).

2. Experimental Procedure

2.1. Raw Materials

Fused alumina powders (75%), graphite flakes (20%) and additives (5%) were used as the basic mixture of the sensor. The chemical compositions of the mixture are presented in **Table 1**. The liquid phenolic resin (8.5%) (Type: 5408, SQhepworth, Yingkou, China) was used as the binder.

2.2. Manufacture of the Sensor

The manufacture process of the sensor is shown in **Fig. 1**. 1. At first, the raw materials were mixed and dried at the temperature of 70°C for 0.5 h. Secondly, the materials were formed at 150 MPa by CIP. Then the formed sensors were heated to 270°C for curing of resin binder. Besides, the cured sensors were heated to a firing temperature and kept for 1 h. The last step was machinery fixing.

2.3. Test of the Porosity and Strength for the Sensor

Strength of the sensor is a key parameter, which is studied by the flexural strength at room temperature after heat treatment. The flexural strength at room temperature is investigated to prevent the sensor from breaking during manufacture, transport and installation. The high temperature flexural strength is an important method to evaluate and estimating the cold flexural strength is only an indirect way primarily. Regarding the research of hot modulus of rupture in carbon containing refractories, there has been a report in which the modulus of rupture was almost constant at high temperature as seen in reference.¹⁷⁾ At this time, the elastic modulus has the linked behavior to the modulus of rupture. And phenolic resin is used as the binder of these refractories, which leads to the similar structure of their binder phase. From above report, it can be presumed that the hot modulus of rupture in this subjected material doesn't show the temperature dependency and has almost constant value. Furthermore, it can also be considered that the ratio of modulus of rupture to elastic modulus, which stands for the resis-

tance for thermal shock, also has a constant value. Hence, the cold flexural strength was evaluated in this study under the condition that heat treatment had been carried out.

The samples were taken from the same position of the cured sensor. Then, they were heated to a firing temperature in the range of 600–950°C at a rate of 2°C/min and kept for 1 h. The flexural strength was measured by three-point loading bending test (crosshead speed: 0.5 mm/min, size of the cuboid sample: 20 × 20 × 100 mm³, span length: 80 mm). The apparent porosity was measured by Archimedes method in distilled water (sample size: 20 × 20 × 20 mm³). Five specimens were used to determine the flexural strength and the apparent porosity.

2.4. Examination of the Binder's Characteristics

Phenolic resin was used to the binder of the Al₂O₃–C refractory, which was heated to 270°C for curing. Then, the changes of weight and decalcescence/heat release of the cured phenolic resin in firing were analyzed by DSC and TG-DTG (NETZSCH STA 409C/CD). The heating rate was 5°C/min from room temperature to 950°C in Ar. The sample was made of 10 mg powders.

The cured phenolic resin was also heated to different temperatures (600–950°C) at 5°C/min and kept for 1 h in order to prepare for the fired binder. An X-ray diffractometer (PW3040/60) that provided Cu K α radiation was used to measure the crystalline-related properties of the fired phenolic resin powders. The step interval was set at 0.02°. The step-scan method was used to determine the *Lc* (stacking height of layer planes) and *La* (a-axis correlation length). *Lc* and *La* were calculated according to the following equation (Scherrer equation):

$$L = k\lambda / \beta \cos\theta \dots\dots\dots (1)$$

where $\lambda = 0.1542$ nm, $k = 0.9$ for *Lc*, $k = 1.84$ for *La* and β is the half-value width (rad) of the X-ray diffraction intensity versus the 2θ curve.^{16,18)}

2.5. Industrial Test for Temperature Response and Lifetime of the Sensor

When the sensor had a good mechanical property and a relatively stable binder structure, the temperature response and lifetime of the sensor would be tested in order to be applied in industrial field. These test works were carried out in Angang Steel Co., Ltd (in Anshan, China). The continu-

Table 1. Chemical compositions of the basic raw materials (in wt.%).

	Al ₂ O ₃	C	CaO	SiO ₂	MgO	Fe ₂ O ₃	Na ₂ O
Fused alumina	99.24	—	—	0.1	—	0.14	0.39
Graphite flake	0.22	97.57	0.13	0.60	0.11	0.28	—

Fig. 1. Manufacture of the Al₂O₃–C sensor.

Fig. 2. Continuous temperature measurement system for molten steel.

Fig. 3. Control test for the sensor's lifetimes.

Table 2. Experimental conditions of the sensor's lifetime test.

Steel grade	Molten steel temperature	Control sensor	
		Response time	Lifetime
SPHC	1550±10°C	140–200 s	24±2 h

ous temperature measurement system for molten steel is shown in Fig. 2. Temperature response was measured as soon as the sensor was inserted into molten steel.

In order to avoid the influences of the different slag layer, molten steel temperature and steel compositions on the sensors' erosion, the lifetimes of the sensor were measured by control trials, as shown in Fig. 3. The sensor fired at 950°C was the control sensor, the positions (A and B) of the experimental sensor and the control sensor would be changed to avoid the influence of the position. The experimental conditions of the sensor's lifetime test are shown in Table 2.

3. Result and Discussion

3.1. Porosity and Strength Variations

Microstructure of the sensor is illustrated in Fig. 4. It can be seen from Fig. 4(a) that an anti-oxidation protective layer about 246 μm uniformly distributed on the sensor's surface preventing the carbon from oxidizing. Fused alumina (FA) and the graphite flakes (G) were bonded by the continuous binder (B) and there was a good dispersion with the aggregates (Fig. 4(b)). Besides, some pores (P) existed in the binder as well as the boundary between the binder and the aggregates (Figs. 4(c) and 4(d)), which would have an adverse effect on the sensor's performances. The binder played the role of the matrix and showed a reticular structure as shown in Fig. 4(d), which was known as glassy carbon and converted from the cured phenolic resin by the firing process. The firing temperature had great influences on the sensor's porosity and strength through the bonded state of the binder phase.

In order to use as the molten steel temperature sensor, it is necessary for $\text{Al}_2\text{O}_3\text{-C}$ refractory to have a high strength, a low porosity and a relatively stable binder through an appropriate firing temperature. Apparent porosity of the $\text{Al}_2\text{O}_3\text{-C}$ sensor at different firing temperatures is shown in Fig. 5. It could be observed that the sensor had a high apparent porosity (9.4%–10.3%) with the firing temperature below 750°C, which is related to the pyrolysis of the binder. The main reaction is the formation of new pores, resulting from the gas generation in this temperature range. In prac-

Fig. 4. Microstructure of the $\text{Al}_2\text{O}_3\text{-C}$ sensor (OM): (FA) fused alumina; (G) graphite; (B) binder; (P) pore.

Fig. 5. Variation of the apparent porosity with the firing temperature.

tice, the chemical densification of the binder, which can lead the porosity to reduce, also exists simultaneously; the degree of this effect is low. As the firing temperature increased to 800°C, the chemical densification greatly improved and resulted in a sharp reducing of the apparent porosity to 8.8%, which is mainly due to the fast shrinkage, repacking and cross-linking of the glassy carbon.¹⁶⁾ With the temperature rising from 800 to 950°C, the sensor exhibited a low apparent porosity about 8.8%–8.4%, which is attributable to the slow shrinkage of the binder above 800°C. Thus, $\text{Al}_2\text{O}_3\text{-C}$ sensor will obtain a low apparent porosity when it is fired under a higher temperature beyond 800°C. This result is well consistent with the Ref. 3).

Figure 6 represents the relationship between the flexural strength of the sensor and the firing temperature. It was found that the firing temperature greatly affected the $\text{Al}_2\text{O}_3\text{-C}$ sensors' flexural strength. At the firing temperature below 750°C, the flexural strength was low and decreased from 7.2 to 6.5 MPa. This result is associated with the fact that the sensor contains lots of pores and the bonded state of the binder is poor. With an increasing of firing temperature from 750 to 800°C, the strength rapidly improved to 8.2 MPa, which related to the sharp reducing of the apparent porosity in this temperature range. The porosity variation was associated with the shrinkage of the binder, which was due to that the crystallite grew obviously with a notable endother-

Fig. 6. Variation of the flexural strength with the firing temperature.

mic reaction occurred. Based on these variations, the heating rate during 750–850°C has to be slow in order to have a relatively stable crystallite structure of the binder and obtain high strength with low porosity. The flexural strength enhanced slowly by 0.3 MPa and kept relatively stable with the increase of temperature in the range of 800–950°C. The variation of the flexural strength is in agreement with the changing of the apparent porosity. In other words, the sensor will have a good strength when the porosity is low. In addition, due to the need of the operation in industry plant, the sensor should usually have a flexural strength more than 7 MPa, which leads the sensor to show stable performances. And this is a statistical result during a long period of time. So the firing temperature of the $\text{Al}_2\text{O}_3\text{-C}$ sensor should be up to 800°C for a low porosity and a high strength.

3.2. Changes of Weight and Decalcence/Heat Release Behavior of the Binder

Mechanism of firing is pyrolysis of the cured phenolic resin which involves complex physical and chemical reactions, such as endothermic and exothermic behavior, weight loss and microstructure evolution. In this paper, the changes of the weight and decalescence/heat release reaction of the binder were analyzed by carrying out the DSC-TG-DTG experiment. **Figures 7 and 8** depict curves of TG-DTG and DSC for the cured phenolic resin in the firing process respectively. When the firing temperature was below 600°C, the main exothermic reaction had finished and the weight of the binder reduced by 80% in total reduction. The weight loss is related to the removing volatiles such as H_2O , phenol and cresol (LMS), CH_4 , C_2H_6 , CO and CO_2 ,^{15,16)} which generates a lot of pores in the binder and the boundary of the aggregates and binder. The strong exothermic reaction also happens with the weight loss and makes the polymer structure gradually transform into the glassy carbon, which result in a high porosity and a low strength of the $\text{Al}_2\text{O}_3\text{-C}$ sensor in this temperature range.

As the temperature increased from 600 to 750°C, the heat release variation was as low as 0.047 mW/mg when the mass decreased by 6.3%. That is exactly the deep pyrolysis of the binder, some new pores also formed. These reactions resulted in an increasing of porosity by 0.9% and the dropping of strength by 0.6 MPa. The heat release variation is almost stable, which is resulted from the fact that the released heat of producing volatiles is almost equal to the

Fig. 7. TG-DTG curve of the cured phenolic resin in firing.

Fig. 8. DSC curve of the cured phenolic resin in firing.

absorbed heat of the chemical densification. The mass merely decreased of 0.5% from 750 to 800°C, but the absorbed heat increased a lot from -0.043 to 0.067 mW/mg. It showed that the absorbed heat of the chemical densification has significantly improved and the shrinkage of the binder is the main reaction, there also rules out a small amount of gases, such as H_2 , CO and CO_2 .¹⁵⁾ These lead to the sharp reducing of the porosity and the rapid increasing of the strength. The binder fired above 800°C showed a slowly steady endothermic reaction and a low mass decreasing. It indicated that the binder is relatively stable and the bonded state is good. Therefore, changes of weight loss and decalescence/heat release behavior are well corresponded to the changes of the porosity and strength.

3.3. Microstructure Evolution of the Binder

Microstructure evolution also exists in the firing process of the binder. The material used for the sensor needs to obtain a binder with a relatively stable amorphous structure through an appropriate firing temperature. The structure evolution of the binder at different firing temperatures was investigated by XRD and Scherrer equation. **Figure 9** shows the X-ray diffraction patterns of the binder while the firing temperature varied from 600 to 950°C. The X-ray diffraction pattern of the binder fired at 600°C showed a broad maximum peak near $2\theta \approx 23^\circ$, it is due to adjacent chains of linear polymer,¹⁶⁾ this structure isn't stable and isn't suitable for the binder's structure. Above 600°C, the broad maximum peak disappeared because of the complete disappearance of the organic polymer structure and the formation of the glassy carbon structure. Another peak is also shown

near $2\theta \approx 43^\circ$ in Fig. 9. Both of the peaks relate to (002) and (10) reflections of carbon materials ((10) contains (100) and (101)). At higher temperatures, the two diffraction patterns gradually increased in intensity, which was related to the lengthening and broadening of the carbon basal planes of the binder's amorphous structure. Meanwhile, the amorphous structure of the glassy carbon gradually becomes stable with the firing temperature increase.

In order to obtain a stable structure for the binder, the crystallite size variation of the binder's amorphous structure with the firing temperature was studied. According to the variation of X-ray diffraction patterns, the crystallite sizes were analyzed by Scherrer Eq. (1). **Figure 10** represents the variations of stacking height of layer planes (L_c) and a-axis correlation length (L_a) with firing temperature. L_c and L_a were small and increased a little by 0.08 and 0.09 nm respectively below 750°C , because the aromatic ribbon molecules of the glassy carbon have started cross-linking and the basal planes enlarged a little by generating polyacene.¹⁵⁾ In

Fig. 9. X-ray diffraction patterns of the binder under different firing temperatures.

Fig. 10. Variations of L_c and L_a of the binder as a function of the firing temperature.

practice, this indicates that the shrinkage of the binder is low and the formation of the new pores is the main reaction due to the elimination of gases, which arouses the reduction of the strength and the increase of the porosity. As the temperature increased from 750 to 800°C , L_c and L_a increased very fast from 0.85 to 0.98 nm and from 1.92 to 2.13 nm respectively, which is due to a drastic endothermic reaction through the following reaction:¹⁶⁾

The occurrence of this reaction acquires the energy by the decalescence behavior, making L_c and L_a increase greatly, which is associated with the rapid enhancement of the endothermic reaction between 750 – 800°C . In detail, the graphitic basal planes grow along the c-axis and a-axis, which results in the rapid shrinkage of the binder. This causes the remarkable reduction of the porosity and the fast increase of the strength. Thus, the heating rate in this temperature range should be slow in order to obtain a finer crystallite structure of the binder phase for a good bonded state. With the increase of the firing temperature from 800 to 950°C , L_c and L_a increased slowly by 0.06 and 0.11 nm, it implied that the amorphous structure remains relatively stable in this temperature range, which is identical with the changes of weight loss and decalescence/heat release reaction of the binder.

3.4. Industrial Application Test for the Sensor's Temperature Response and Lifetime

In order to be applied in industrial operation, the Al_2O_3 –C sensor should have a high strength, a low porosity and a relatively stable binder. On the basis of above discussion, when the firing temperature is up to 800°C , the sensor has good mechanical properties, which has been confirmed by the mentioned experiment and mechanism research. Therefore, the present work studied the service properties (temperature response and lifetime) of the sensor fired at 800°C (called the experimental sensor) for industrial application. 10 sensors (No.: 8000–8009) fired at 800°C and 10 control sensors (No.: 9500–9509) fired at 950°C were used for the industrial test.

It can be seen from **Fig. 11** that the temperature response curve of the experimental sensor is monotonous increase without fluctuation. And the response time of the experimental sensor was 165 s, which was almost equal to the control sensor about 140 – 200 s. In practice, the firing temperature

Fig. 11. Temperature response curve of the experimental sensor.

Table 3. Lifetimes of the sensors fired at 800 and 950°C.

No.	Lifetime (sensor A): h	Lifetime (sensor B): h	Steel grade
1	25.3 (8000)	24.5 (9500)	SPHC
2	22.5 (8001)	22.0 (9501)	SPHC
3	24.5 (9502)	23.5 (8002)	SPHC
4	25.0 (9503)	24.3 (8003)	SPHC
5	25.0 (8004)	24.5 (9504)	SPHC
6	23.6 (8005)	23.5 (9505)	SPHC
7	24.2 (9506)	23.2 (8006)	SPHC
8	24.5 (9507)	24.3 (8007)	SPHC
9	25.3 (8008)	24.5 (9508)	SPHC
10	23.7 (9509)	22.4 (8009)	SPHC

affects the thermal conductivity of the sensor through the porosity and the binder's structure. It means that the firing temperatures (800 and 950°C) have no remarkable influence on the heat transfer performance of the sensor in temperature response, which is associated with that the sensors have almost the same porosity and binder's amorphous structure. Thus, the temperature response of the experimental sensor is satisfied with the temperature measurement for molten steel.

As shown in **Table 3**, the experimental sensor and the control sensor had similar lifetimes in the same continuous casting conditions that the steel grade was SPHC and the casting temperature was about 1550°C. The average lifetimes of the sensors fired by 800 and 950°C were 23.94 and 24.09 h respectively, the deviation of lifetimes was below 1.5 h (about 6%) in the same experiment. These showed that the erosion rates of the sensors are similar, which is due to the experimental sensor has similar porosity, strength and binder's structure as the control sensor. The deviation of lifetimes in the same experiment comes from the little difference of the sensors' positions (position A or B). The sensor B has a little more serious erosion due to be adjacent to the long nozzle. Besides, the lifetimes with the same position and steel grade vary a little on the dependence of the difference in actual continuous casting conditions, such as the molten steel temperature fluctuation and the slag layer thickness variation. All of these indicated that the sensor fired at 800°C has almost the same lifetime with the sensor fired at 950°C, which is well consistent with the changes of the strength, porosity and binder's characteristics. The temperature 800°C is a suitable firing temperature for good performances of the $\text{Al}_2\text{O}_3\text{-C}$ sensor, which has higher efficiency and lower energy consumption compared to the present firing temperature.

4. Conclusion

The effect of firing temperature on the mechanical prop-

erties of $\text{Al}_2\text{O}_3\text{-C}$ system material, which is used for continuous temperature measurement sensor of molten steel, was investigated in this study. And the mechanism of this effect was investigated to obtain a suitable firing temperature. In addition, the temperature response and the lifetime of the sensor with the suitable firing temperature were tested by an industrial application. The main results are as follows.

(1) The $\text{Al}_2\text{O}_3\text{-C}$ sensor had a low strength (7.2–6.5 MPa) and a high porosity (9.4–10.3%) when the firing temperature was at 600 to 750°C. The strength rapidly increased from 6.5 to 8.2 MPa and the porosity sharply decreased from 10.3% to 8.8% during 750–800°C. Strengths and porosities of the sensors fired under higher temperature up to 950°C were similar (8.2–8.5 MPa and 8.8%–8.4% respectively).

(2) The binder's main exothermic reaction and weight loss occurred below 750°C, and then caused many pores. Due to the sharply enhanced endothermic reaction, L_c and L_a increased greatly from 750 to 800°C, which led to the binder's rapid shrinkage. The binder had slow changes of the endothermic reaction and crystallite size between 800–950°C, which caused its relatively stable amorphous structure.

(3) Response time and lifetime of the sensor fired at 800°C were almost equal to the sensor fired at 950°C during continuous casting of SPHC steel, which were about 165 s and 24 h.

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