Note

Tracking Softening-Melting Behaviour of Blast Furnace Burden

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The softening and melting properties of ferrous materials are controlled by reduction degree, basicity, amount of gangue and flux, phase chemistry and their distribution in the microstructure. Design of laboratory softening-melting tests simulating blast furnace conditions greatly affects the reduction degree of the ferrous materials, since heating rate and reduction gas profile are key parameters affecting reduction degree of burden. Softening-melting behaviors of different blast furnace burden were compared by running laboratory tests under modified conditions. Comparing conventional and modified softening-melting test conditions, it can be said that modified test conditions are more practical adaptation of blast furnace conditions. SEM analysis indicated the primary mechanism for softening is due to presence of wustite as primary liquid slag former under modified test conditions, which is more close to blast furnace situation.

KEY WORDS: softening; melting; slag; reduction degree.

1. Introduction

The cohesive zone inside blast furnace, where the ferrous burden softens and melts, significantly affects the productivity of the blast furnace.¹⁾ The deformation of the ferrous burden unit is the macroscopic result of softening, the final step is the final meltdown.²⁾ Considerable gas pressure drop occurs in the cohesive zone; requiring the softening temperatures of ferrous burdens to be high and the difference between softening and melting points minimal.³⁾ The softening and melting properties of ferrous materials are controlled by reduction degree, basicity, amount of gangue and flux, phase chemistry and their distribution in the microstructure. The reduction degree refers to the fraction of oxygen removed from the ferrous oxides.

Design of laboratory softening-melting tests simulating blast furnace conditions greatly affects the reduction degree of the ferrous materials. Heating rate and reduction gas profile are key parameters affecting reduction degree of burden. While ‘Conventional’ softening-melting test is currently being conducted under fixed gas composition till complete melting of the test portion, ‘Modified’ softening-melting can be conducted applying varying gas composition with temperature/time to simulate the ideal condition, as the

burden would have experienced inside blast furnace. In the present scope of work, such modified tests were attempted to track the softening-melting behavior of blast furnace burden inside customized blast furnace conditions.

2. Experimental

Conventional softening-melting tests involve subjecting a bed of mixed burden of 90 mm height used as a sample portion taken in a graphite crucible of 48 mm diameter with perforated top and bottom support for passage of reducing gas.⁴⁾ Reducing gas mix is used at a flow rate of 6 liters per minute. Softening-melting behavior of burden mix is normally characterized by: softening temperature (T_s), temperature in °C at which the pressure differential across the bed is 100 mmWC (mm Water Column); melting temperature (T_m), temperature in °C at which the pressure drop across bed again is back to 100 mmWC; ΔP_{max} , the maximum pressure differential across the bed in mmWC (as shown in Fig. 2(a)). Conventional softening-melting test has a sample temperature profile as shown in Fig. 1(a) following a heating cycle with almost no thermal reserve zone. The reducing gas composition (70% N_2 + 30% CO) is kept fixed (Fig. 1(c)) during entire test corresponds to wustite/iron equilibrium condition, without any CO_2 . This amounts to simulation of upper stack region of blast furnace with a much rich gas than practically available.

Whereas, modified softening-melting test uses a typical reducing gas profile as shown in Fig. 1(d) below, estimated using theoretical considerations. Reduction gas used in modified test was considered as mixture of CO, CO_2 and N_2 . Modified softening-melting test also has a sample temperature profile with typical thermal reserve zone adopted from ideal blast furnace temperature profile, which is quite different from that used in conventional softening-melting test, as shown in Fig. 1(b). 100% sinter (super-fluxed) and mix of 70% sinter & 30% iron-ore as used in blast furnace was subjected to both conventional and modified softening-melting test for comparing softening-melting behaviour. Chemical composition of the raw materials used are shown in Table 1. These tests were further repeated and quenched at different stages of softening-melting test [start of softening (T_s), attaining maximum pressure drop (ΔP_{max}) just before dripping—refer Fig. 2] to generate samples for in-depth analysis with microstructural analysis.

3. Results and Discussions

Above 900°C, rise in temperature is quite slow in modified test in comparison to conventional softening-melting test and gas is also leaner resulting lower reduction degree. Calculated degree of reduction at T_s in modified test, by balancing oxygen between inlet and outlet gas, was around 40% which is significantly lower than the degree of reduction of around 60% observed in conventional test. This probably results an early formation of primary slag with presence of sufficient amount of wustite as liquid phase former and early softening of the mix (Fig. 2). Due to slower heating rate coupled with early softening resulting prolonged residence time of semi-solid slag bearing fractions, maximum pressure drop and overall pressure drop

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Fig. 1. Conventional & Modified softening-melting test: (a,b) temperature-time plot (c,d) reducing gas mixture-temperature plot.

Table 1. Chemical composition of raw materials.

	Fe (t), %	CaO, %	SiO ₂ , %	P, %	MgO, %	MnO, %	Al ₂ O ₃ , %	TiO ₂ , %	FeO, %	K ₂ O, %
Sinter	57.41	9.15	4.27	0.093	1.7	0.061	2.36	0.21	10.06	0.057
Ore	63.11	0.21	1.06	0.064	0.098	0.11	3.34	0.31	—	—

Fig. 2. Bed pressure drop with 75% sinter+30% ore in (a) Conventional, (b) Modified softening-melting test.

(area under the curve) observed was also higher for modified test in comparison to conventional softening-melting test. Effectively the sinter sample exhibited broad/thicker softening-melting zone in modified softening-melting test than conventional softening-melting test.

The quenched samples (at T_s and ΔP_{max}) were mounted and polished in resin for study in reflected light. The samples were subjected to SEM-EDS analysis. SEM analysis was done by JEOL-SEM. The results of the analysis are shown in Table 2 below for quenched samples with 100%

sinter and in Table 3 for quenched samples with 70% sinter & 30% ore. Six and five distinct phases were observed in samples quenched at T_s (start of softening) for 100% sinter and sinter-ore mix respectively. Whereas, no. of phases reduced to 2–3 for samples quenched at ΔP_{max} , just before dripping and remaining phases were not present as indicated in the tables. The identification of phases were done using XRD analysis and confirmed with SEM analysis. Elemental composition as reported from SEM analysis is based on point analysis (average of 10–15 individual data points) of phases present and hence might not represent exact composition but can be used for qualitative interpretation. Due to non-stoichiometric composition, the phases detected could not be identified as particular mineral types. It can be seen from the table that more no. of phases (mostly calcium-silicate with different partitioning of FeO, Al₂O₃ and MgO) are present in slag of samples quenched at T_s . With further rise in temperature and close to liquidus temperature (sample quenched at ΔP_{max} , just before dripping), slag became leaner in FeO and richer in other gangue and flux bearing components. Also, no. of phases at higher temperature reduced, indicating complete formation of nearly homogeneous primary slag assimilating the available fluxes.

Presence of different types of slag phases for 100% sinter sample quenched at T_s was shown in Fig. 3(a). The bright parts were Fe-rich whereas grey parts were slag. Presence of magnetite, metal and slag phase (for 100% sinter sample quenched at ΔP_{max}) and formation of metal phase from magnetite was clearly visible in Fig. 3(b). The neck formation and interconnectivity of metallic phase was observed in metal phase for sample quenched at ΔP_{max} . The amount of slag phase also decreased with reference to samples quenched at T_s .

Table 2. List of slag phases and their composition in quenched test samples (for 100% sinter).

PHASES present	Modified softening-melting test quenched at Ts (with 100% sinter)					Modified softening-melting test quenched at $\Delta P_{\max}$ (with 100% sinter)				
	% elemental composition					% elemental composition				
	Fe	Ca	Si	Al	Mg	Fe	Ca	Si	Al	Mg
Slag phase										
Phase 1	52.60	23.05	11.28	11.26	1.82			Not present		
Phase 2	28.29	34.82	17.56	17.74	1.59			Not present		
Phase 3	46.34	25.52	24.17	0.54	3.44	45.81	10.06	6.29	16.98	20.86
Phase- 4	35.29	27.60	3.29	31.75	2.07			Not present		
Phase- 5	9.36	82.20	7.77	0.22	0.45	0.23	60.83	28.53	4.13	6.28
Phase- 6			Not present			0	44.61	17.86	35.82	1.70

Table 3. List of slag phases and their composition in quenched test samples (for 70% sinter + 30% ore).

PHASES present	Modified softening-melting test quenched at Ts (with 70% sinter + 30% ore)					Modified softening-melting test quenched at $\Delta P_{\max}$ (with 70% sinter + 30% ore)				
	% elemental composition					% elemental composition				
	Fe	Ca	Si	Al	Mg	Fe	Ca	Si	Al	Mg
Slag phase										
Phase 1	62.26	12.98	17.14	7.56	0.06	84.08	7.69	4.46	1.66	2.12
Phase 2	13.39	39.97	23.44	22.10	0.89	9.86	38.91	18.35	27.98	4.90
Phase 3	45.34	0	31.59	21.98	1.10			Not present		
Phase 4	55.10	1.44	1.45	41.32	0			Not present		
Phase 5	15.58	68.39	10.61	2.85	2.57			Not present

Fig. 3. SEM Pictures of quenched samples of 100% sinter during modified softening-melting test at (a) Ts, (b) $\Delta P_{\max}$.

Similar broad observations were also noted for SEM analysis with quenched samples (at Ts and $\Delta P_{\max}$) with mix of 70% sinter and 30% ore. For mixed burden samples, calcium-silicate phases in slag of sample quenched at Ts, were observed with high Al content (alumino-silicate, alumino-ferrite). These are contributed by high alumina

content of iron-ore in mixed burden. MgO content in slag was higher for 100% sinter burden than mixed burden due to higher MgO input through sinter.

4. Conclusion

Softening-melting behaviors of different blast furnace burden were studied in detail by running laboratory tests under modified conditions. Comparing conventional and modified softening-melting test conditions, it can be said that modified test conditions are more practical adaptation of blast furnace conditions. Softening mechanisms under modified conditions were further analyzed using microstructural (SEM) study of samples generated from quenched tests at two different points of softening-melting zone (Ts and $\Delta P_{\max}$). SEM analysis indicated the primary mechanism for softening was due to presence of wustite as primary liquid slag former under modified test conditions which is more close to blast furnace situation. Therefore, careful selection of laboratory test conditions by correlating results of modified softening-melting tests with burden performance in actual blast furnace in future, can markedly improve the accuracy and practical utility of the softening-melting test results.

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