LADLE SANDS: TESTING AND APPLICATION

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INTRODUCTION

The increasing demand for better and cleaner steels and the increase in melt shop productivity has changed the way steel is made and delivered to continuous casters. These changes result in higher steel temperatures and longer holding times, both of which adversely affect ladle free opening rates.

For a smooth caster operation, the rate of ladle free opening is ideally 100%. In order to achieve this, the well in the pocket block is filled with sand prior to tapping. This sand prevents the molten steel from freezing in the well block, thereby blocking the nozzle opening. If the sand does not flow freely, then the nozzle is burned open with an oxygen lance. This creates the need to temporarily remove the ladle shroud and allows the metal to be exposed to air which is an undesirable practice as it causes the deterioration of the steel’s cleanliness.

In 1984, the Refractories Research Group at Armco started to investigate and develop test procedures to evaluate ladle sands before they were tried in the field. The laboratory work concentrated on determining the properties a sand should have in order to maximize its probability of permitting a ladle free opening to occur. Once a determination was made of what constitutes a "good" sand, other elements like method of sand placement, sand temperature, length of holding time, etc., were tracked in the field.

This paper will report on the development of a test procedure to evaluate sands and discuss the results obtained in the laboratory and in the field.

THEORY OF LADLE SANDS

The optimum sand for use to fill the ladle well block is one which develops a thinly sintered layer on the hot face, i.e., next to the steel, which is in a semi-liquid state while at temperature. Once the slide gate is opened, the majority of the sand flows out and the ferrostatic pressure exerted by the steel in the ladle breaks the sintered layer. A slight sintering of the sand is necessary to prevent the grains from washing into the steel during ladle fill or subsequent refining steps. Theoretical considerations show that as steel temperatures increase and are added to long holding times, they provide the conditions necessary for an increase in the thickness of this sintered layer. Due to the steep thermal gradient encountered in this area, the sintered layer can start to crystallize near the cold face becoming very strong and forming a barrier to the steel flow.

Sand grain sizing affects the maximum packing density that can be achieved. Improper sizing distribution leads to poor packing which allows for steel penetration between the grains and prevents the metal from breaking through at the appropriate time. A large amount of fines in the sand increases the surface area available for sintering thus causing it to become strongly sintered and preventing steel flow initiation. Impurities, like free silica in zircon sand, can add to this problem by lowering its refractoriness.

LITERATURE REVIEW

In the last few years, several papers have been presented that describe problems with ladle free openings, steps taken to improve the situation and reasons for the problem. One of the main parameters which affect ladle free openings is the sand used in the well block. Depending on the type of sand investigated, these papers can be divided into: silica (SiO₂), zircon (ZrSiO₄), or chromite. Kq and Chiên¹, Chiên et al.², and Pan and Ko³ extensively investigated the reactions which occur when high purity silica sands mixed with small amounts of alkali feldspar (microcline: KAlSi₃O₈) are used to fill the ladle block well. They concluded that the main parameters of concern when selecting a sand were alkali content, sand particle shape and purity. During field trials, the effect of holding time and steel temperature in relation to differing silica to feldspar ratios were noted as they affected ladle free open. Shiotani, et al.⁴, developed a test procedure that could be carried out in the laboratory before the sand was tried in the field. The only sands investigated were of silica composition with small additions of alkali feldspar. Their work agreed with the previously mentioned studies in that they found that high purity silica sand with a small amount of alkali gave the best results in the field. In addition, size distribution of the grains was determined to be an important property. The pit practices investigated were sand fill method, inside shape of well block and well block refractory. Wessel, et al.⁵, discussed the role of impurities associated with pure zircon sand. In this case, they determined that the maximum acceptable amount of
staurolite \(\text{Fe}_2\text{Al}_2\text{O}_7(\text{SiO}_4)\text{OH}\), a zircon sand impurity, had to be less than 500 ppm for optimum free opening rate. Additional parameters investigated dealt with optimum well design, pit practices, finish-argon-rinse-to-ladle-open time and end-of-tap-to-ladle-open time. By implementing changes in sand chemistry and operating conditions, they were able to improve the free opening rate from about 77% to 96%. The paper by Garlick and Lucas\(^5\) is the only one that mentions the use of chromite sands with carbon additions. They also investigated silica sands. Their work consisted of determining the sintering behavior and thermal conductivity of these sands. This information was used to formulate a simple heat transfer model which incorporated the effects of ladle temperature and steel holding time on sand behavior. Garlick and Lucas\(^5\) concluded that the most desirable sand should have a low thermal conductivity and high sintering temperature so as to minimize the thickness of the sintered layer.

**BACKGROUND**

In the mid 80's, several of the Armco steel plants were experiencing poor free opening rates. One plant was as poor as 30%, while another was at 70%. As these numbers were unacceptable for the operation of the affected steel shops, a laboratory investigation was started to ascertain if and how sand properties affect the ladle free opening rate. Once it was established that the sand's characteristics played a major role, work was initiated to determine which kind of sand would have the highest probability of consistently providing ladle free opening. To this end, laboratory test procedures were developed to serve as predictors of sands that provide reliable performance before they are used in the field.

**TEST PROCEDURES**

A sand submitted to the laboratory for evaluation is subjected to several tests each one more severe than the previous one. This ensures that the more difficult or time consuming tests are only carried out on candidates likely to succeed.

The first test, and the one all sands undergo, consists of two parts: a screen analysis of the grains and a sintering test. The choice of sieves used for the screen analysis was dictated by the sizes available in the laboratory, those commonly used in the raw materials industry and the sizes which provided the best packing density. In order to fulfill all of these requirements, sieve sizes had to be changed during the study. The currently used sizes are +80, +100, +140, +200 and pan.

The sintering test consists of filling a brick crucible with a measured amount of sand. The gap between the top of the sand and the lid is filled with carbon to create a reducing atmosphere. The assemblage is then placed in a furnace where it is fired to 1600°C (2900°F) and kept at temperature for four hours. The four hour hold was chosen because it mimics one of the most severe applications the sand will see in any of Armco's plants. After the firing and subsequent cooling, the sand is carefully removed from the crucible and graded as to its degree of sintering. The grading scale is from 1 to 5, with 1 being the best and 5 the worst. The following list describes the rating procedure:

If a sand is rated a "1", this means that no sintering has occurred and the sand remains granular.

If a sand is rated a "2", this means that enough sintering has occurred so that the sand will hold its shape but, if the sintered slug is rubbed with the fingers, the grains will come loose. The slug can also be crushed by hand.

If a sand is rated a "3", this means that the surface of the sand is sintered hard, but it is only a very thin layer; the interior of the slug is still a "2". This slug can still be crushed by hand.

If a sand is rated a "4", this means that the sand slug is sintered all the way through, but the grains can be removed when scraped with a sharp object like a screwdriver.

If a sand is rated a "5", this means that the slug is totally sintered and no amount of scraping will remove grains.

If the sand passes, i.e., scores lower than a 2.5 in the above rating, then it is sent for a chemical analysis. The sand is analyzed for all major oxides, alkalis, loss on ignition and for free silica as distinct from total silica. A special test procedure had to be developed for a direct free silica analysis. Work carried out early in this study showed that the free silica was concentrated in the 200 mesh and smaller grain sizing.

The last test performed on the sand is a metal interaction test. It closely simulates operating conditions taken to an extreme. The test set-up consists of a crucible, "the ladle," sitting on top of a well block with a slide gate plate attached to it (Figure 1). The well block hole is filled with a predetermined amount of dried sand. The crucible and sand are heated with a gas burner to operating temperatures. Argon gas is purged into the apparatus to rid it of excess oxygen. Steel scrap of appropriate chemistry is charged into the crucible, melted on top of the sand and kept in contact with it for a predetermined length of time. Time and temperature are adjusted.
Fig. 1. Metal/sand test set-up.

to resemble the most extreme conditions encountered at the particular melt shop for which this work is being carried out. After the allotted time has elapsed, the slide gate is opened to allow the unsintered sand to fall out. This sand is collected and weighed to determine the percent of unsintered sintered sand. The crucible is cooled and the metal and sand left inside is removed for further studies under the optical and scanning electron microscopes.

DISCUSSION OF LABORATORY RESULTS

Most test series involved the evaluation of zircon samples of different provenance. Nevertheless, other materials like magnesia (MgO), chrome ore and alumina (Al₂O₃) have also been investigated. In some instances, the effect of adding carbon, or increasing the purity of the mix was studied. Table I summarizes the sand data that will be discussed in this paper, but many more sands than those mentioned in this table have been evaluated in the laboratory. Microscopy work was carried out on the Australian and Floridian zircon sands and the chrome ore + zircon sand because these sands were of interest for field application or were already in use in the field.

Both the Australian and the Floridian zircon sands are naturally occurring minerals which are mined and shipped with no beneficiation or grain sizing taking place. Grains are well rounded and few impurities are present. The magnesia and alumina grains are man-made products which are available in a range of chemistries and grain sizing. Because they have to be crushed or ground in order to achieve the desired sizing, the grains tend to be very angular.

Two different varieties of magnesia grains were investigated. The grains which contained 5% free silica showed borderline characteristics in the sinterability test as shown by a rating of 2.5 (Table I). The high purity magnesia passed the sinterability test (rating = 1), but failed the metal test. As it is predominantly composed of one grain size (Table I), the packing density was poor leading to a considerable amount of voids which filled with metal while running the metal sand test. Changes in grain sizing were not investigated as that would have made the product too expensive for field use.

Alumina grains are routinely used to prevent brick from sticking to each other during high temperature laboratory testing. Two grain compositions were tested as to their applicability for use in improving ladle free opening. The 95.5% Al₂O₃ grain did not pass the sinterability test (rating = 4), but the higher purity alumina grain did (rating = 1). Upon further testing, the high purity grain failed the metal test because it fused and reacted with the metal.

Testing of chrome ore with and without the addition of carbon showed it to fail the sinterability test (ratings of 4 and 5 respectively).

One of the most commonly used sands in the steel industry is zircon. This mineral can be purchased containing different levels of impurities which reflect its geographical source. For this study, we investigated zircon sands from Australia and Florida, USA. The main differences between these sands can be found in the level of free silica and in the sizing distribution. The Australian sand is coarser than the Floridian one and it has minimal free silica (Table I). When subjected to the sinterability test, the Australian sand was only slightly sintered (rating = 2) while the Floridian sand was almost completely sintered (rating = 4). The addition of carbon to this latter sand slightly improved its sinterability (rating = 3). The Australian sand, and the Floridian sand with the carbon addition, were both subjected to the metal interaction test. The former performed well while the latter failed. The Floridian zircon sand with the carbon addition was subjected to the metal test because it was being used in the field at that time.

The zircon sand plus chrome ore was added to the test series because it is a sand which performs well in the field, although it failed the sinterability test. Its free silica level is very low as its only source is the zircon component of the mix. The screen analysis shows that each component has its unique particle size with the coarser fraction representing mainly chrome ore.

MICROSCOPY

The Australian sand is characterized by an extremely low free silica level. Figure 2 is a typical view of this sand in the as-received condition. A small grain of silica can be seen among the zircons. These grains constitute the free silica in Table I. Heating this sand to 1600°C causes the zircon grains to become weakly bonded at temperature. Figure 3 shows zircon grains chained
### Table I

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<th>Sand type</th>
<th>AUSTRALIAN/FLORIDIAN</th>
<th>ZRCON</th>
<th>ZIRCON</th>
<th>FLORIDIAN</th>
<th>ZRCON+O</th>
<th>CHROME ORE</th>
<th>CHROME ORE + C</th>
<th>ZIRCON + MgO GRAIN</th>
<th>MgO GRAIN</th>
<th>AI2O3 GRAIN</th>
<th>HIGH PURITY</th>
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**Sinterability (1600°C)**

| 4 Hour Hold | 2 | 4 | 3 | 4 | 5 | 4 | 2.5 | 1 | 4 | 1 |

(See Comments)

**Metal/Sand Test**

(1600°C for 4 hrs.)

| Steel penet. | Steel penet. | Penet. was
<table>
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<tr>
<td>Penet. was</td>
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<tr>
<td>7mm; 25%</td>
<td>7mm; 81%</td>
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<td>2.5-3.5 mm</td>
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**COMMENTS:**

1. No sintering
2. Slight sintering
3. Thin sintered layer
4. Sintered, but some grains can be removed
5. Totally sintered

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**Fig. 2.** Photomicrograph of a typical view of the Australian zircon sand in the as-received condition (Z=zircon, S=silica). Magnification: 115X

**Fig. 3.** Photomicrograph of the Australian zircon sand after heating to 1600°C for 4 hours. Note the thin silica bridges holding the zircon grains together. A secondary phase is also found in the silica (Z=zircon, S=silica, SP=silica containing secondary phase). Magnification: 230X
together by means of thin glassy bridges. The composition of these bridges or glue is silica as determined by scanning electron microscopy. Sometimes, a secondary phase can also be found in these glassy areas. The source of the glass is the melting of the free silica grains and the decomposition of zircon into zirconia and silica (Figure 4). Investigation of the phase diagram of the system ZrO$_2$-SiO$_2$ shows confliction melting and dissociation temperatures depending on the amount of impurities present$^{7,8,9}$. If impurities are present the decomposition can start at temperatures as low as 1350°C, while in a pure system, it happens at 1540°C.

![Fig. 4. Photomicrograph of the Australian zircon sand showing the decomposition of zircon into zirconia and silica (Z=zircon, S= silica, ZA=zirconia). Magnification: 230X](image)

The Floridian zircon sand grains are generally smaller and more angular than the Australian ones. Table I shows the size distribution and Figure 5 illustrates the morphology of this sand in the as-received condition. At high temperatures, the same bonding mechanism is in effect as for the Australian sand. Figure 6 is a view of a silica grain bonding three zircon grains. With time at temperature, the bond area will look more like the section shown in Figure 7. Here, dendrites within the bonding silica have scavenged the impurities found in the sand creating complex silicates containing Ca, Al, and Ti. These impurities also have the effect of increasing the amount of zircon grains that revert to zirconia plus silica which adds to the amount of silica available for bonding.

![Fig. 5. Photomicrograph of a typical view of the Floridian zircon sand in the as-received condition (Z=zircon, S=silica). Magnification: 115X](image)

The zircon plus chrome ore sand is characterized by big, angular chromite pieces interspaced with small, rounded zircon grains

![Fig. 6. Photomicrograph of the Floridian zircon sand after heating to 1600°C for 4 hours showing a silica grain bonding three zircon grains (Z=zircon, S=silica). Magnification: 230X](image)

(Figure 8). Once this mixture has been heated, complex structures develop (Figure 9). This photomicrograph shows the intimate contact the chromite and the zircon/zirconia grains attain. The silicate serves as a glue to hold everything together. Figure 10 shows a similar area, but observed through the scanning electron microscope. In this case, the zircon has partially dissociated into zirconia; the chromite grain has remained unaffected by the heat and a complex silicate surrounds the grains and fills all interstices. This silicate was a liquid at temperature as can be deduced by Figure 11 which shows secondary crystallization of zircon overgrowths onto zirconia and also primary and secondary chromites.

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FIELD WORK

A great number of sands used in the United States are of the zircon type. For that reason, most of the work was done with the aim to determine the optimum properties for this kind of sand. As a result of the microscopy work and the determination of the grain size fraction containing the free silica, a specification was written incorporating this information. In addition, the total amount of allowable free silica was determined to be less than 0.2%. A grain sizing component was also added to the above specification. The implementation of this specification allowed us to improve the ladle free opening rate from 33 to 80% in one case.

To achieve further improvements, changes in the method of sand emplacement, sand dryness and ladle well block clean out procedures had to be addressed. The physical layout of the melt shop made it difficult to put the sand into the well block. The operator found himself behind a building column, had to stand very close to the preheated ladle and could not see where the sand had to go. In order to fix this problem, a sanding station was built which allowed the operator to observe where the sand was placed. The station was far enough removed from the preheated ladle that the heat from it was not
overwhelming. The station was of sufficient size so that sand could be stored on it. A specially heated hopper was designed to keep the sand moisture free and at a uniform temperature. If a suspicion should arise that the grain sizing was not as specified, a set of sieves is kept on the platform so that an immediate check can be run. To achieve a consistent well block fill, the amount of sand put into the ladle is carefully measured. Another area that was addressed was the cleaning of the well block. New procedures were instituted to ensure a thorough and consistent cleaning.

The biggest operational effect on ladle free opening performance was found to be the steel residence time as measured from end-of-argon stir to beginning-of-cast. The longer the argon-to-cast time, the lower the probability of a ladle free opening (Figure 12). As this was a continuing problem in the field, additional sand compositions were tried. The most successful one in decreasing the number of non-openers was zircon sand mechanically mixed with chrome ore. The use of this sand has brought the free opening rate to 95% or better, depending on the type of steel. An additional advantage was that the zircon/chrome mixture was lower in cost compared to pure zircon. A problem which has been encountered since this study was carried out is that heats held for more than three hours from tap-to-cast still have an erratic free opening rate.

DISCUSSION AND CONCLUSIONS

The Australian zircon sand used for this study is a prime example of a material that fulfills all the criteria for a good sand.

In the laboratory tests, it showed good packing characteristics, no fines fraction, minimal impurities and very slight sintering. In field tests, it performed well, although some of the improvements can be attributed to changes in operating and pit practices. Microscopy work is described for the weak intergranular sinter as very thin silica bridges glued the zircon/zirconia grains together. This silica is probably semi-liquid at temperature as deduced from the morphology of the crystals growing in it.

The successful field use of zircon sand mixed with chrome ore indicates that the sinterability test may not be applicable to all sand compositions as this mixture failed it. Laboratory work showed this sand to have good packing density and also contain a small fraction of fines. The microscopy study of the sintered sand indicates that the bonding agent is liquid at steelmaking temperatures, and its quantity is a function of the length of time spent at that temperature. The failure of this sand to work appropriately when long holding times are involved is probably due to the chrome ore as it contains low melting point impurities. This leads to an increase in the thickness of the sintered layer which, due to the thermal gradient in this part of a steel ladle, will start recrystallizing and become too strong to be broken by the ferrostatic pressure of the steel.

As a result of the work carried out, the optimum ladle sand should sinter weakly at the sand/steel interface and the sintered layer should remain thin over time. The sand properties which ensure these results are dependent on its grain sizing distribution, the amount of impurities present and its chemistry. A test procedure was developed to ensure that the behavior of the sand is appropriate for the application before it is put into service.
REFERENCES


