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# Dilatometric Characterization of Direct Reduction Pellets

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**ABSTRACT**

During the reduction process, the increase in volume of the iron ore pellets leads to a decrease in the mechanical resistance or disintegration of the pellets which lead to the unfeasibility of the process. The present work has the objective of evaluating the thermal expansion behavior of the direct reduction pellets using dilatometry. For this, the dilatometric expansion curve and the coefficient of thermal expansion of the pellets were determined through a horizontal pushrod dilatometer under an argon atmosphere. The dilatometric results showed that the expansion of the pellet increased and the degree of expansion decreased as the temperature increased, and changes in the expansion curve demonstrated the occurrence of microstructural transformations due to the presence of MgO and $SiO_2$ in the composition of the pellet. The pellets were submitted to thermal cycles with a gradual increase of temperature up to 600°C, 900°C, 1000°C and 1050°C, which presented similar results of coefficient of expansion and made it possible to relate the dilatometric results with the compressive strength of the pellets, which intensified with material expansion to the temperature of 1000°C and showed significant loss of strength when exhibited shrinkage in the temperature range of 1000-1050°C.

***Keywords –*** Dilatometry, Coefficient of thermal expansion, Compressive strength, Pellets, Thermal expansion.

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## I. INTRODUCTION

Dilatometry is a thermoanalytical technique where the length variations of a sample are measured as a function of temperature or time, while the sample is subjected to a specific atmosphere under heating, cooling, or even under isothermal conditions, i.e. it is a method by which the thermal expansion of a material can be measured [1].

The results of the dilatometry are presented as dilatometric curves $dl/l_0$ as a function of T (variation of length based on the initial length as a function of temperature) [1-3]. An accurate analysis of the dilatometric curve allows to understand the thermal behavior of several materials, such as the phase transformations that are accompanied by the dimensional change of the material and observed at the inflection points of the curve. In addition, the dilatometry is well applied to determine the coefficient of linear thermal expansion of the material in each temperature range [4].

The coefficient of linear thermal expansion is a property of the material and expresses the degree to which the material expands or retracts with changes in temperature. This property is widely used to investigate the dimensional behavior of composite structures of different materials, which may fail when subjected to temperature changes or thermal stresses [3].

Materials such as direct reduction pellets are subjected to different temperatures since loaded onto the top of the reduction furnace until pass through the reduction zone. During the reduction process, it is observed the occurrence of swelling phenomenon, which consists in the increase of volume of the pellets due to the changes in the crystalline structure. The swelling is associated with loss of resistance or disintegration of the pellets and considered to be catastrophic if the swelling exceeds 20%, as it impairs the permeability of the furnace with the generation of fine particles [5-7].

Thus, pellet swelling behavior was studied under different conditions: pellet characteristics (chemical composition, microstructure and porosity), firing parameters (temperature and time), reduction parameters (temperature, time and gas composition) [8-11].

Due to the importance of dilatometry as a different method to evaluate the thermal behavior of materials subjected to high temperature, the present work has the objective of evaluating the expansion behavior of the direct reduction pellets using dilatometry. For this, the work focuses on the evaluation of the dilatometric curve and the determination of the coefficient of thermal expansion of pellets that pass through the pelletizing process. The study was performed under argon atmosphere in a horizontal pushrod dilatometer, where different pellets were submitted to a gradual increase of temperature up to 600°C, 900°C, 1000°C and 1050°C, to verify the effectiveness in the application of this different technique and to evaluate the relation of the dilatometric results with the compressive strength of the pellets.

## II. MATERIALS AND METHODS

### 2.1 Materials

Direct reduction pellets used in this work were produced by the pelletizing process. Pellet size was on average 12 mm in diameter, 2.8 g in weight with average compressive strength of 950 N/pellet. As seen from Fig. 1, the X-ray diffraction result shows that the pellet is mainly composed of the hematite phase ($Fe_2O_3$) and small amount of magnetite ($Fe_3O_4$).

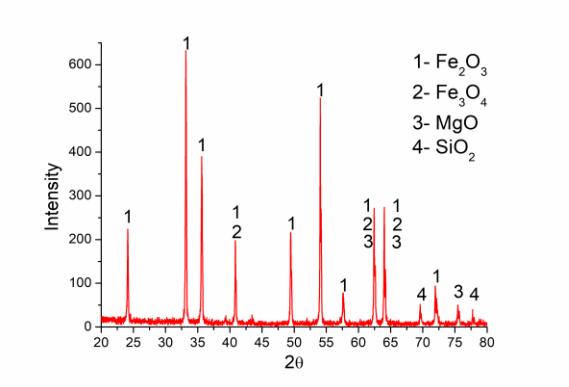


Fig. 1. X-ray diffraction of the direct reduction pellet.

As demonstrated in Fig. 1, the presence of MgO and $SiO_2$ were also detected in the phase composition of the pellet. S. Dwarapudi et al. [9] evaluated the effect of MgO on the swelling behavior of the hematite pellets during the reduction and concluded that the addition of MgO considerably reduces the tendency of pellets to swell due to the formation of high melting point slag that gives sufficient bond strength to withstand the reduction stresses.

### 2.2 Dilatometry

To evaluate the thermal behavior of the direct reduction pellets, they were submitted to tests in the Dil 402C horizontal pushrod type dilatometer.

The dilatometer is composed of a sample holder inside the oven, which is applicable to a temperature range of -180°C to 2000°C and a linear variable displacement transducer (LVDT) with a maximum measurement range of 5000 µm. To perform the tests, in addition to the dilatometer, the measurement system consists of a controller of the thermal analysis system (TASC) 414/4, which connects the hardware of the dilatometer to the measurement software; the power supply of the furnace; thermostat, which has the function of maintaining the LVDT with a constant temperature of 25°C and a computer system for recording and processing the data.

In the experiment, the thermal cycle of 0-1050°C was divided into 4 heating programs for the dilatometric tests of pellets RD01, RD02, RD03 and RD04. The heating program corresponds to a successive increase of the temperature to be reached by each pellet within a temperature range with a defined heating rate, according to Table 1.

Table 1. Heating program.

| Pellets | Temperature range °C | Heating rate °C/min |
|---|---|---|
| RD01 | 0-600 | 7 |
| RD02 | 600-900 | 1,73 |
| RD03 | 900-1000 | 0,68 |
| RD04 | 1000-1050 | 0,23 |

Each sample was tested until it reached a different temperature range to evaluate the application of dilatometry with analysis of the dilatometric results in the same temperature ranges. To perform the process in the dilatometer, the tests with the pellets were carried out under the argon atmosphere with flow of 50 ml/min.

### 2.3 Compressive strength

The direct reduction pellets were individually subjected to a compression test to evaluate the pellet strength after dilatometry testing at different temperatures and its relation to the dilatometric results. The tests were performed in the EMIC model DL-60 machine with a load cell with a rated capacity of 20KN and test speed of 10mm/min.

The pellets were placed between two parallel plates and submitted to compression until their fracture. The mechanical strength of the pellets was expressed in N/pellet.

## III. RESULTS AND DISCUSSION

### 3.1 Linear thermal expansion / Thermal expansion coefficient

The thermal expansion results ($dl/l_0$/% x Temperatura/°C) of the pellets RD01, RD02, RD03 and RD04 are respectively shown in Fig. 2 (a-d).

In Fig. 2 (a), it is observed that the pellet RD01, tested up to 600°C, presents a linear dilatometric curve, characterizing only the influence of temperature on the variation of sample length. In the Fig. 2 (b-d), this behavior is verified in the expansion curves of the pellets RD02, RD03 and RD04, which is proved by the results of the coefficient of thermal expansion, evaluated in the temperature range between 100-600°C, as shown in Table 2 (a-d).

As presented in Fig. 2 (b), with the test advance up to 900°C, the thermal expansion curve of the pellet RD02 exhibits a small slope increase from the temperature of 600°C, which is also verified in the curves of pellets RD03 and RD04, as shown in Fig. 2 (c-d). These pellets have practically the same degree of expansion in the temperature range of 600-900°C as shown in Table 2 (b-d), which proves the same change in the expansion curve.

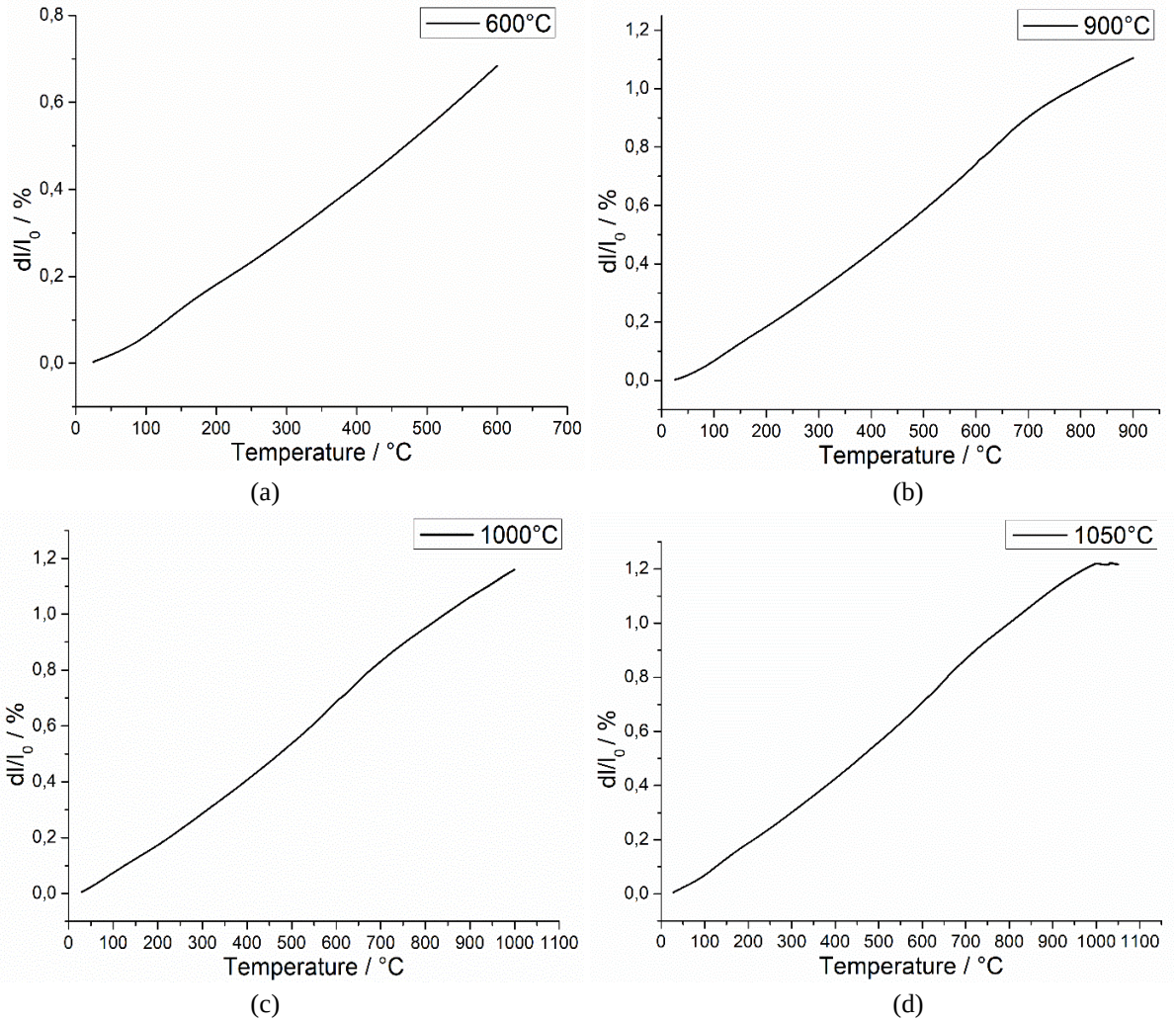


Fig. 2. Thermal expansion of the pellets: (a) RD01, (b) RD02, (c) RD03 and (d) RD04.

The pellet RD03 was tested to the temperature of 1000°C and shows a decrease in the slope of the dilatometric curve in the temperature range of 900-1000°C, as shown in Fig. 2 (c). In Fig. 2 (d), it is observed similar behavior in the expansion curve of pellet RD04, which is justified by the decrease in the degree of thermal expansion in the temperature range 900-1000°C, as shown in Table 2 (c-d).

The dilatometric curve of the RD04 pellet exhibits all thermal expansion behavior with the test performed up to the temperature of 1050°C, as presented in Fig. 2 (d). After reaching 1000°C, the expansive behavior of the pellet RD04 is followed by a contraction, just in the range of 1000-1050°C, where the degree of thermal expansion decreases significantly, as shown in Table 2 (d). This decrease in the degree of expansion demonstrates that microstructural transformations occurred and another relation of coefficient of expansion with temperature was obtained.

Table 2. Coefficient of thermal expansion of the pellets: (a) RD01, (b) RD02, (c) RD03 and (d) RD04.

| | Pellets | α(100-600°C) | α(600-900°C) | α(900-1000°C) | α(1000-1050°C) |
|---|---|---|---|---|---|
| (a) | RD01 | 1,23E-05/°C | | | |
| (b) | RD02 | 1,25E-05/°C | 1,21E-05/°C | | |
| (c) | RD03 | 1,22E-05/°C | 1,25E-05/°C | 0,98E-05/°C | |
| (d) | RD04 | 1,27E-05/°C | 1,20E-05/°C | 0,98E-05/°C | -1,93E-05/°C |

The dilatometric analysis progressively, raising the temperature in each test, allowed to verify the same thermal expansion behavior of the different pellets as shown, which were confirmed in the results of coefficient of thermal expansion measured in the same temperature ranges. This result, in addition to showing that the dilatometry can be used as a different method for thermal analysis of the pellets, shows that the phase compositions of the 4 pellets are identical, that is, i.e. pellets are composed mainly of hematite ($Fe_2O_3$), as illustrated in Fig. 1.

With the completion of the thermal cycle, it is verified that the dilatometric result of the direct reduction pellet, composed of hematite ($Fe_2O_3$), showed an increase of its dimensional variation as the temperature was increased, according to Fig. 2 (d).

**3.2 Analysis of microstructural transformations**

The result in Fig. 1 showed the presence of compounds such as MgO e $SiO_2$ in phase composition of the pellet. Meyer and Lu apud M.M. Machado [12] report that during the process of thermal treatment of the pellets, several chemical reactions occur due to the composition of the pellets and the temperature applied. The authors report the occurrence of a reaction between hematite ($Fe_2O_3$) and magnesium oxide (MgO) forming the ferrite magnesium ($MgO.Fe_2O_3$) of cubic structure in a temperature range of 450 to 900°C. Another reaction occurs between the MgO and $SiO_2$ compounds forming the magnesium silicate ($2MgO.SiO_2$) of orthorhombic structure at a temperature from 1000°C.

According to S. J. Casarini [4], microstructural transformations are observed at the inflection points of the dilatometric curve. In Fig. 2 (b-d), it is observed that the inflection of the dilatometric curve of the pellets RD02, RD03 and RD04 occurs between the temperatures of 600°C and 900°C due to formation of the ferrite magnesium ($MgO.Fe_2O_3$). The curve of the pellet RD04 in Fig. 2 (d) exhibits a contraction behavior after 1000°C, which comprises the formation of the magnesium silicate ($2MgO.SiO_2$) due to the reaction between the MgO and $SiO_2$ compounds from this temperature.

**3.3 Compressive strength**

In the pelletizing process, the pellets are subjected to a gas flow with a temperature of about 1350°C in the firing step. In this step, the occurrence of sintering reactions between the iron ore particles and formation of partially liquid phases provides the pellets mechanical resistance to withstand the thermal and mechanical stresses within the reduction furnace [13].

The result of the compressive strength related to test temperature of pellets RD01, RD02, RD03 and RD04 is shown in Fig. 3.

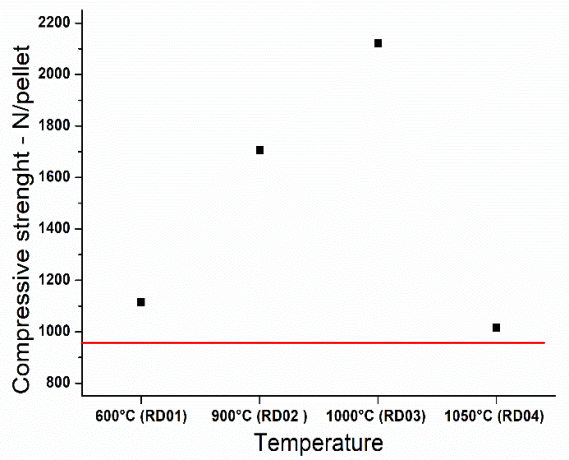


Fig. 3. Relation between the compressive strength and the test temperature of pellets RD01, RD02, RD03 and RD04.

It is observed that with successive increase of the test temperature of the pellets, the compressive strength increased to 1000°C. This result shows that the complementary heating under argon atmosphere up to 1000°C, a continuity of the reactions that occur in the firing step provided an increase in the compressive strength up to 1000°C. With the test, up to 1050°C, the resistance fell sharply and reached a lower value than the result found at 600°C, as shown in Fig. 3.

The increase of the compressive strength up to 1000°C accompanied the continuous character of pellets expansion in the temperature ranges between 100-600°C, 600-900°C and 900-1000°C, as shown in Table 2 (a-c). However, with the contraction behavior in the temperature range 1000-1050°C in Table 2 (d), the pellet exhibited a decrease in its strength at 1050°C, as shown in Fig. 3.

## IV. CONCLUSION

- The dilatometric curves of the pellets showed practically the same expansion behavior when comparing the same temperature ranges from one test to the other. This pattern of results, confirmed by the results of expansion coefficients, shows uniformity in the phase composition of the pellets and validates the dilatometry as an adequate technique in the evaluation of the thermal behavior of the pellets.
- The dilatometric results of the pellets composed mainly of hematite showed that with the increase of temperature, the dimensional variation of the pellets increases while the degree of expansion presents an inverse tendency.
- Changes in the expansion curve in the temperature ranges between 600-900°C and 1000-1050°C are attributed to the microstructural transformations due to the presence of MgO and $SiO_2$ compounds in the pellets and heating under the argon atmosphere.
- The correlation of the dilatometric results with the compressive strength shows that the increased resistance of the pellet increased along with their expansion, however after exhibiting contraction behavior, the pellet resistance decreases significantly.

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