ENERGY BALANCE OF THE CARBOTHERMIC REDUCTION OF IRON ORE ENHANCED BY MICROWAVE ENERGY[1]

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Abstract

With the objective to perform a high confidence determination of rate of reaction and energy balance of carbothermic reduction of iron ore was developed a microwave assisted reduction reactor, with full control and measurement of irradiated microwave power and measurement of reflected microwave power and actual weight of sample during the reducing process. This reactor permits to monitor the rate of reduction and the equipment losses of energy. For the startup of this new equipment was done a series of iron ore reductions to establish a basic procedure of operation and identify its malleability and possibilities of use. Spherical pellets with weight of 3,5g and diameter of 15 mm were prepared with Carajás iron ore and coke in stoichiometric proportion. They were irradiated with 1kW of 2.45 GHz microwaves. There were obtained curves of reaction tax, power evolution during the reduction and global energy balances.

Key words: Iron carbothermic reduction; Microwave assisted reduction; Energy balance.

BALANÇO DE ENERGIA EM REDUÇÃO CARBOTÉRMICA DE MINÉRIO DE FERRO AQUECIDA POR MICROONDAS

Resumo

Com a idéia de realizar balanços de energia e determinação de taxa de reação com altos níveis de confiança, foi desenvolvido um forno para redução carbotérmica aquecido por microondas com controle e medida da potência irradiada e medida da potência refletida e efetiva, e de massa da amostra durante o período de processamento. Este reator permite monitorar a taxa de redução e a perda de energia do sistema. De início, realizou-se uma série de reduções de minério de ferro Carajás para estabelecer o procedimento básico de operação deste equipamento inovador, identificando sua maleabilidade e possibilidades de uso. Pelotas esféricas com massa de 3,5g e diâmetro de 15 mm foram preparadas com minério de ferro de Carajás e coque. Elas foram irradiadas com 1000 W de microondas de 2,45 GHz. Foram obtidas curvas da taxa de reação, evolução da potência durante a redução e balanço global de energia.

Palavras-chave: Redução carbotérmica; Redução aquecida por microondas; Balanço de energia.

1 *Technical contribution to the 7th Japan-Brazil Symposium on Dust Processin-Energy-Environment in Metallurgical Industries and 1st International Seminar on Self-reducing and Cold Bold Agglomeration, September 8-10 2008, São Paulo City – São Paulo State – Brazil*

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1 INTRODUCTION

Another application of microwave technology has been tested in various metallurgical processes such as the measure of the thickness and composition of the slag in an induction furnace and analyses of gases. However, the use of microwave energy in carbothermic reduction has been the most attractive theme in microwaves applied the metallurgy. Many works were focused on showing the comparison between the rate of reduction of metal oxides by conventional methods and by microwave enhanced reduction, in special the benefits of shorter processing time.

Recently some works are given special attention to the environment, as microwave enhanced recycling of Zn, Ni, Cr and Pb present in the dust generated in the manufacture of iron and steel.

The state of art shows us that the equipments utilized for carbothermic reductions are basically domestic microwave ovens adapted to perform these reactions. This improvisation was good for initial works, where the objective was to explore a new subject and prove the possibility of the microwaves use for reduction of metallic oxides. Nowadays, microwave enhanced carbothermic reduction of metallic oxides is an accepted reality, but not completely explained.

At this moment, more accurate data on energy balance and reaction tax are required, the adapted domestic ovens becomes insufficient to produce the needed information, for example the effective microwave power absorbed by reacting materials, the real mass decrease of reactants during the reduction time. To help the microwave enhanced carbothermic reduction to grow to a well established science, was developed the equipment described in this paper.

2 MATERIALS AND METHODS

2.1 The Microwave Furnace for Mineral Oxides Reduction

This furnace was developed at Instituto Mauá de Tecnologia and has two important sections, the microwave one and the reduction chamber. Figure 1 shows an overview of this equipment and figure 2 its schematic representation.

Microwaves are generated by a microwave variable-power generator of 2.45 GHz protected by a circulator. Two directional couplers and two power meters measure the microwave transmitted power and the reflected power. A moving short cut move the point of maximum electric field to a position over the pellet.

Microwave filters open safely the access to the inner chamber of irradiation where is located the pellet. Through down side filter are introduced the pellet and the ceramic/Teflon support, and by the upper side the reaction fumes are discharged. The down side filter is connected to a bag where argon flows at 10 L/min. In the bag is located a balance to monitor the weight loss of pellet. The inner temperature of reduction chamber may be read by an infrared pyrometer, through a mirror. The wave guides are wrapped in heat exchangers to cool them during the reduction process. The heat exchanger has thermometers in the inlet and outlet of refrigeration water. The flow of argon and water was asserted by rotameters.

Figure 1 shows an overview of the equipment utilized for carbothermic reduction with microwaves

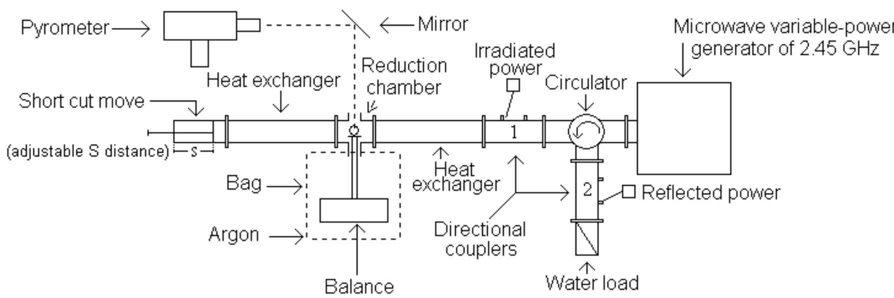


Figure 2 shows the schematic representation of the equipment utilized for carbothermic reduction with microwaves

2.2 Searching for Operational Procedures

To establish how to proceed there was done over several tests to identify the better pellet diameter, the better flow of argon and the better crucible shape.

This was a trial and error step. It was started with pellet of 18 mm without crucible. In this case, electrical arc was observed in several tests, just when the reduction was over. Then the pellet becomes protected by a crucible. Figure 3 shows two pellets, one without a crucible and other with an open crucible, booths on the microwave cavity support for pellets.

Figure 3 shows two pellets, one without a crucible and other with an open crucible

The crucible was prepared from microwave transparent ceramic fiber. They imposed a reduction in pellet diameter to 15 mm. With this crucible the number of arc occurrence was reduced, but not eliminated. Then the crucible received a cap Figure 4 shows a crucible and its caps, also from ceramic fibers.

Figure 4 shows a crucible and its caps, also from ceramic fibers

With this cap the arcs were completely avoided, but several crucibles melted at the end of reduction. Figure 5 shows a melted crucible. This problem was solved with increase of the argon flow until it was enough strong to cool the crucible avoiding its melting.

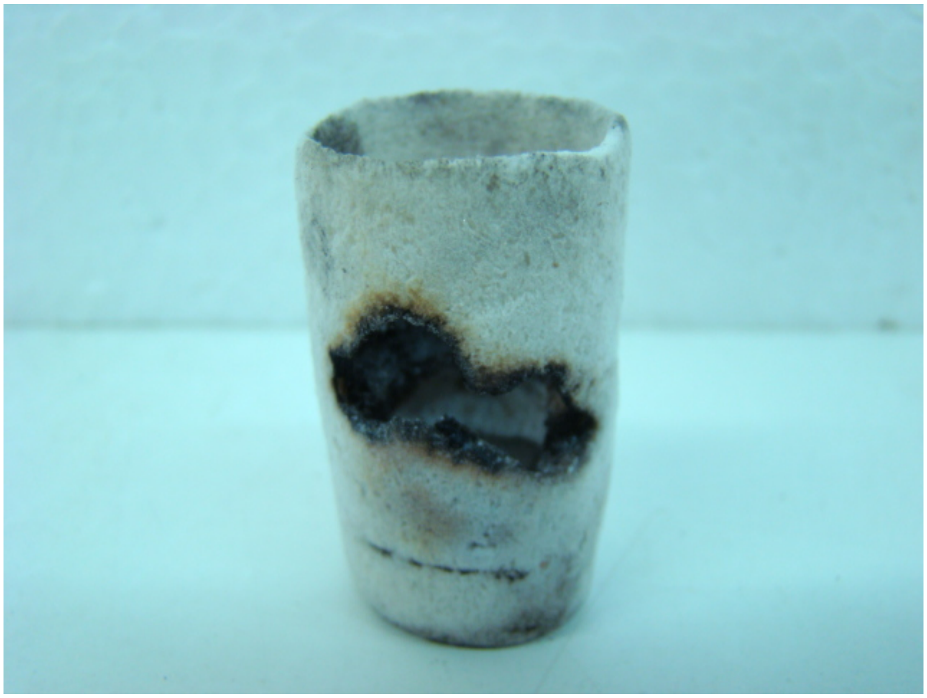
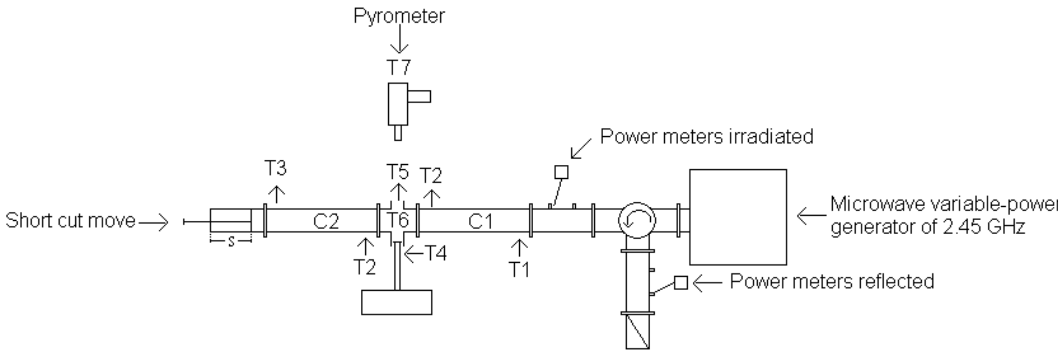


Figure 5 shows a melted crucible

2.3 Reduction and Power Curves and Energy Balance

Weight loss, incident and reflected power data was digitally acquired. Data on weight versus time and power measurements were mathematically treated to produce curves of reaction tax and power evolution.

The energy balance was based on temperature and fluxes data collected by T1, T2, T3 thermometers, water flow rotameters, argon flowmeters and treated to determine the effective energy loss both the flow of water and argon gas were kept constant throughout the test at a rate of 0.5 L / min and 10 L / min, respectively.



T1: Temperature of the cooling water entrance the changer heat C1;
T2: Temperature of the cooling water exit and entrance the changer heat C1 and C2, respectively;
T3: Temperature of the cooling water exit the changer heat C2;
T4: Temperature of argon gas entrance in the reactor;
T5: Temperature of argon gas and gas generated in the reactor;
T6: Surface temperature in the reactor;
T7: Surface temperature in the pellet;
C1: Changer heat 1;
C2: Changer heat 2;

Figure 6 shows the schematic diagram of the experimental apparatus for determination energy balance

The global energy balance compute the energy supplied to the reactor, the reactor energy and the losses of energy by the system.

The energy supplied to the reduction system is determined empirically measuring the irradiated and reflected microwave power, as shown in equation 1 and 2.

$$E_{provided\ the\ process} = \theta \times P_{effective} \quad (1)$$

Where $E\,(kJ)$ energy supplied; $\theta\,(s)$ processing time; $P_{effective}\,(J/s)$ effective microwave power determined in equation 2.

$$P_{effective} = P_{irradiated} - P_{reflected} \quad (2)$$

Where $P_{irradiated}\,(J/s)$ irradiated microwave power; $P_{reflected}\,(J/s)$ reflected microwave power.

The overall equation for the energy balance is:

$$E_{provided\ the\ process} = E_{provided\ the\ reaction} + E_{loss\ in\ reactor} \quad (3)$$

Where $E_{provided\ process}$ is calculated by equation 1; $E_{provided\ the\ reaction}$ is the energy supplied to the reactants and it is calculated through equation 4; and $E_{loss\ in\ reactor}$ is the energy losted by reduction system and it is calculated through equation 5.

$$E_{reactor} = m_{Fe_2O_3} \times \Delta H_{reaction} + m_{pellet} \times cp \times \Delta T \quad (4)$$

$$E_{loss\ in\ reactor} = [m \times cp \times (\Delta T_{C1} + \Delta T_{C2})] + (m \times cp \times \Delta T)_{pellet} + (k_1 \times A \times \Delta T)_{Ar} + [\Delta T / (L/k_2)]_{CR} \quad (5)$$

Where L is thickness and k_1 and k_2 is thermal conductivity in argon and chamber of reaction (CR), respectively.

All data for equation 3 maybe obtained experimentally. With these data, it is possible to verify the energy distribution during carbothermic reduction.

3 RESULTS AND DISCUSSION

3.1 Operational Procedures

Carajás iron ore was analyzed for iron content by permanganometry and petroleum coke was analyzed for fixed carbon content by ASTM D3172-89 method. With this contents was formulated a stoichiometric compositions for deducing iron ore with carboxiethylcellulose as the binder. Dried pellets was reduced to a 15 mm diameter pellet and weighted.

The weighed pellet was charged in the covered crucible, which was then placed on top ceramic/Teflon support and inserted inside the reactor through the microwave filters and supported on a semi-analytical balance to monitor the pellet mass loss. The pellet position in the chamber of reaction is always the middle of the reduction cavity. The circulation of cooling water was started with a flow of 0.5 L/min. Also the flow of inert gas dry argon inside the reactor to 10L/min. The pellet was irradiated with 1000W of 2,45 GHz microwaves. The effective and reflected powers were recorded. Each 2 minutes the temperatures of the cooling system as the surface of the pellet were monitorated. The microwave irradiation was done until the loss weight becomes null and the reduced pellet start to gain weight by its oxidation. The reduced pellet was cooled to room temperature by the flow of argon.

1.2 Reduction and Power Curves

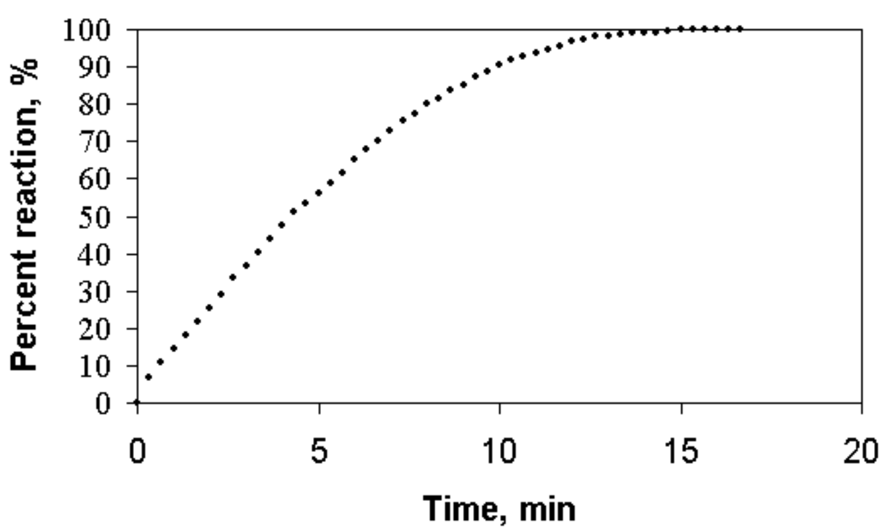


Figure 7 shows the reaction tax

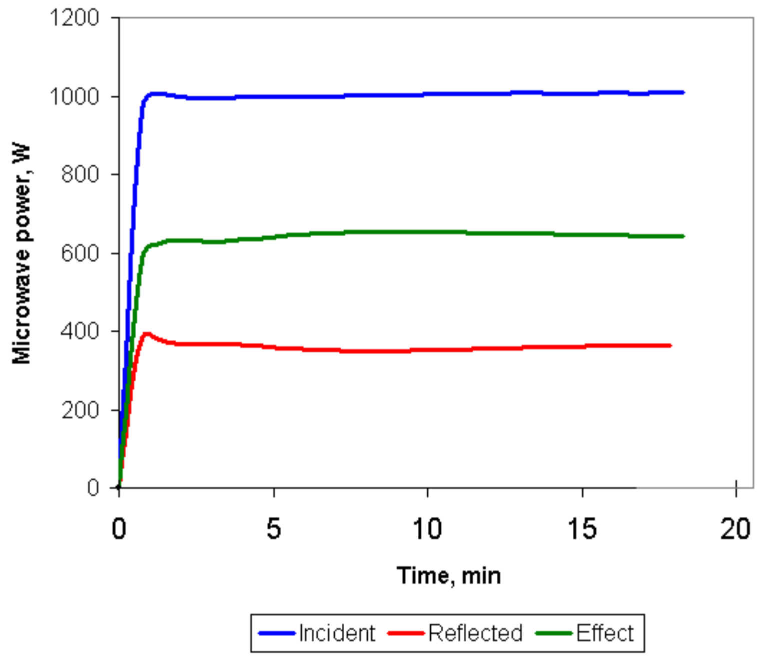


Figure 8 shows the power evolution during the reduction

The average curve of reaction tax for six repetitions is shown in figure 7. It is possible to see that there was a complete reduction of iron ore by petroleum coke in the stoichiometric mixture prepared, in 15 minutes of reaction. Figure 8 shows the power evolution during the reduction. It is possible to see that the is radiated power, that is fed to the cavity, is kept practically constant by the microwave generator, close to the set point of 1000W. The reflected power, which is the parcel of the radiated power that was not absorbed by the system, and effective power, which is the difference between irradiated and reflected powers, are practically constant. It is possible to affirm that the behaviour of the iron/coke system is uniform during all time of reduction.

1.3 Energy Balance

Table 1 presents the data for energy balance

		Test 1	Test 2	Test 3	Test 4	Test 5	Test 6
$E_{losses}\,(kJ)$	C1	2,51	5,86	4,19	7,54	8,37	7,54
	C2	36,84	43,54	41,87	46,89	39,77	38,52
	Gas	615,5	624,0	630,29	619,2	614,8	621,47
	Reactor	2,08	2,09	2,08	2,08	2,09	2,08
$E_{reaction}\,(kJ)$	$\Delta H_{reaction}$	6,71					
	Total	663,64	682,18	685,14	682,42	671,74	676,32
$E_{effective}\,(kJ)$		775,1	774,0	760,9	782,5	738,4	699,5
	Difference	111,46	91,82	75,76	100,08	66,66	23,18

4 CONCLUSIONS

The experiment conducted has shown that it is possible to specify and accurately reproduce the conditions of irradiation by microwave energy for the analysis of the system, in a process of reduction carbothermic stimulate by microwave.

The good results obtained for Carajás iron ore reduction with stoichiometric petroleum coke under 2,45 GHz microwave irradiation permit us to conclude that the equipment described in this paper reproduce the results of usual experiments of microwave aided carbothermic reduction. More than reproduce tax reactions this new equipment permit to control and measure the effective energy transferred to the reaction system.

This equipment allows us to analyze the energy distribution in the reduction system. It shows the energy losses thought equipment by conductive and radiation heating, the heat loss by argon flow and the effective energy allowed for the carbothermic reduction reaction.

The are some operational resorts which will be introduced in a future model, for example a direct pellet temperature reading and off gas analyses.

Acknowledgement

The authors would like to thank L. F. Baccan and R. B. Nascimento for technical support. Support from Instituto Mauá de Tecnologia and to the São Paulo State Foundation for Research Support (FAPESP) is gratefully acknowledged.

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