

NEW TRENDS IN BLAST FURNACE COKE CHARACTERIZATION

by D. VOGT* - J.M. DUCHENE * - J.M. STEILER ** - C. THIRION ***

presented by C. MELTZHEIM ***

SUMMARY

The regularity and good working order of the blast furnace are determined by total control of the permeability of the burden. In this respect, size analysis of the coke constitutes an essential factor. New information concerning its behavior in the blast furnace has been deduced by direct observations using blast furnace dissections and probes. In this perspective, probing equipment through the tuyeres has recently been installed at SOLMER with the collaboration of IRSID. The coke is subjected to intense granulometric degradation between the throat and the tuyeres. This deterioration can not be entirely predicted by conventional cold testing techniques ; in fact, it also depends on the thermal and chemical history of the coke in the blast furnace as well as the stress to which it is subjected.

The purpose of this study is to analyze the different aspects of the coke's physico-chemical behavior in relation to the production parameters such as the nature of the blends and the carbonization conditions.

An examination of cokes produced in a pilot oven using blends which cover a wide range of ranks has allowed us to quantify the binary interactions between the coals during carbonization and to demonstrate the influence of the physico-chemical characteristics of these cokes.

These characteristics have been evaluated by different laboratory tests, especially the gasification test developed by the "Centre de Pyrolyse de MARIENAU" and IRSID, which simulates the blast furnace. A quantitative morphological characterization of the coke carbon phase of the textures (anisotropy) has been carried out using a texture analyzer.

The essential parameters which govern the behavior of the coke during gasification have been specified :

- the coal rank which, along with the carbonization conditions, determines the coke texture,
- the mineralogy of the ashes (basicity, alkalines, etc.) which influence the kinetics of the CO₂ gasification,
- the characteristics of the porous network.

(*) "Centre de Pyrolyse de MARIENAU"

(**) IRSID - "Institut de Recherche de la Sidérurgie"

(***) SOLMER

These parameters were taken into account in the development of methods for forecasting the coke reaction rate.

Within the predictable context of greater fluctuations in the quality of coals received, these techniques of anticipating coke characteristics have proved to be a useful tool for mastering the quality of the coke charged in the blast furnace.

I - INTRODUCTION

Direct observations concerning the evolution of blast furnace coke characteristics are now relatively abundant and agree with the results of the studies.

Two complementary approaches have been made in recent years : sectionings of quenched blast furnaces and probings with industrial apparatus.

The main contribution of blast furnace sectionings, carried out especially in Japan, has been to provide a 3-dimensional picture of the internal condition of the blast furnace and point out the essential role of the melting zone. Observations concerning coke have shown considerable degradation of its characteristics in the high temperature zones, especially near the tuyeres, the center of intense thermal, mechanical and chemical stress.

The samplings using coke taken from the tuyeres of industrial blast furnaces, carried out on a large scale in Europe between 1975 and 1980, confirmed the dissection results in regards to coke degradation in the blast furnace, but more importantly showed that this degradation could not be considered independently of the blast furnace operating conditions.

A supplementary step in this direction, adding to our knowledge of blast furnace hearth phenomena, has recently been taken with the installation of a tuyere probing system by IRSID and SOLMER. This process takes coke for sampling along the radius and up to the center of the blast furnace. We will describe this equipment briefly and then present some typical results for illustrating our arguments concerning the degradation of coke characteristics between the throat and the tuyeres.

II. - TUYERE PROBING OF THE SOLMER BLAST FURNACE

1. Characteristics of the machine

This machine was installed at SOLMER at the end of 1984 on blast furnace n° 2 (production : 6,000 tons per day, hearth diameter 11.2 meters). A diagram of the machine is provided in figure 1. Two distinct types of tests are carried out :

- when the blast furnace is shut down, a 300 mm diameter core boring up to the axis, with the blow pipe and tuyere removed (fig.2),
- with the blast furnace working, probings with a cooled 90 mm diameter probe through the blow pipe and tuyere up to the core. Different measurements can then be begun (fig.3).

Up to the present time,

- 11 coke borings during blast furnace stoppages and
- more than 700 probings during operation of the blast furnace. have been carried out.

2. Main results

In the present report, we will treat the following four aspects :

- size analysis of the coke
- mechanical strength characteristics
- chemical composition
- microstructure.

1. Size analysis (Figure 4)

Changes in size analysis distribution of the coke have been studied along the radius of the blast furnace. First of all, we have noted an overall decrease in particle size, the mean sizes descending, for example, from 65 mm at the throat to approximately 40 mm at the tuyeres.

Moreover, there is a considerable change in the size distribution along the radius : at the center of the blast furnace, in the dead man zone, the coke pieces are relatively large (35 % > 40 mm) up to approximately 1.3 meters at the tuyere. At this level, the > 40 mm fraction practically disappears and the quantity of fines < 3 mm increases suddenly up to nearly 20 % (in the 0 - 10 mm fraction). This accumulation of fines at the bottom of the tuyeres' raceway indicates the intensity of the stress that the coke is subjected to in this zone.

2. Mechanical strength

The cold mechanical strength of the coke, measured by the % > 10 mm after 600 drum revolutions (1 Drum) is presented in function of the position along the radius. At the center of the blast furnace, the coke shows great strength, similar to the value that it had when charged at the throat. This value decreases the closer one gets to the tuyeres, reaching its minimum near the bottom of the raceway ; this change is completely compatible with the quantity of fines mentioned above.

3. Chemical composition

In regards to its chemical composition, the coke appears to be thoroughly changed after passing in the blast furnace. The "crude" ash content of the coke exceeds 25 % at certain points in the radius, especially beyond the raceway ; a more precise examination shows, however, that this increase in ash

content is, for a large part, due to the penetration of slag and pig iron in the periphery of the pieces of coke. A complete calculation of the different substances and of the "real" coke ash content, i.e. not counting the influence of the pig iron and slag, shows that this value, which is generally higher than at the time of charging, changes very little along the radius. On the other hand, the chemical constitution of the coke ash changes radically between the tuyeres and the throat. The most spectacular result is the change in the alkali content of the ash as shown in figure 6. Within the dead man zone, the alkalines can be the most abundant component after the silica. The K2O content of the ash is approximately 20 %. Near the tuyeres, however, the coke contains very little alkali ; in certain cases there is even less than in the charging coke. This change along the radius points out the importance of the alkaline circulation phenomena, the alkalines being greatly influenced by the temperature : the zone around the tuyeres, subjected to high temperatures, provides conditions which are favourable to the complete vaporization of the alkalines.

4. Microstructure

The last aspect to be considered regarding the changes in the coke between the throat and the tuyeres is presented in figure 7. Here we can see the porous structure of the coke. The coke porosity variations between the throat and the tuyeres of different French blast furnaces have been represented using tuyere coke samples. A generally low rate of change in porosity has been observed (several percentage points). However, this evolution also seems to depend on the nature of the coke. In any case, it does not take the gasification of the coke in the blast furnace into account, the average rate of which is approximately 25 %.

In order to summarize these different observations concerning the evolution of coke in the blast furnace, the following elements will be considered :

- a considerable decrease in the average sizes between the throat and the tuyeres, with an important change along the radius :
 - . relatively large sizes at the center
 - . a great quantity of fines near the bottom of the raceway.
- mechanical strength values which have suffered little except in the immediate vicinity of the raceway.
- considerable chemical alteration with a very great enrichment in alkalines especially in the dead man zone.

- a porous structure at the core of the pieces showing little difference from the structure observed upon charging.

All of these results point towards the strong interaction which exists between the mechanical behavior of the coke and the physical-chemical stress that it is subjected to in the blast furnace :

- high temperature
- partial gasification
- reactivity of the alkalines

These aspects should thus be taken into account for the coke qualification tests. We should also investigate how it is possible to obtain coke which resists these diverse stresses, using the production parameters (coal blends, carbonization).

III. - MEASUREMENT OF SIGNIFICANT CHARACTERISTICS FOR THE BLAST FURNACE

We will distinguish between three series of measurements of coke characteristics : the mechanical tests, the reactivity measurements and the textural characteristics.

1. Mechanical tests

The mechanical tests concern resistance to abrasion and fragmentation. In this regard, the MICUM, IRSID or JIS indexes are now well known.

2. Reactivity measurements

The reactivity measurements have become more common. Their principle and consequently their operating methods vary. We will now compare the two methods which will be used in what follows : "CSR" and "CPM/IRSID" (figure 9).

The CPM/IRSID test is carried out in the following way : 400 grams of coke is heated to 620 to 1,200 °C with a blast furnace type gas (60 % N₂, 10 % H₂, 20 % CO, 10 % CO₂). The test stops at 25 % weight loss. The gasification speed and the gasification threshold temperature are then measured (figure 10). The Japanese test uses 200 grams of coke maintained at 1,100 °C under 100 % CO₂ for 2 hours. The rate of gasification is then measured. The two tests also differ in the mechanical strength measurement after gasification : CPM abrasion test (less than 3 mm), Japanese fragmentation test (more than 10 mm).

3. Structural characteristics (figure 8)

Different methods are used for describing the textures of coke.

Optical microscopy using polarized light and an image analyzer measures the size and the morphology of the anisotropic units of the coke as well as its porosity.

The optically isotropic particles can be studied using transmitted electron microscopy (Oberlin method). The size of the molecular orientations can then be measured.

X-ray diffraction provides an average measurement of the size of the crystallite (thickness of line 002, width of line 100) and of the

inter-reticular spacing \bar{d}
002.

These three complementary methods allow for describing the degree of organization of the aromatic carbonaceous matrix of the coke on all scales.

We will thus study the parameters which can modify this texture as well as its effects on the qualitative criteria directly connected to the operation of the blast furnace such as mechanical strength or reactivity.

IV - PARAMETERS CONNECTED TO THE STRUCTURAL CHARACTERISTICS OF THE COKE

In figure n° 12 we can see the relationships between an essential parameter of the coal blend, the rank (represented by the Vitrinite Reflectance, Ro) and the quality criteria such as reactivity and abrasivity. In both cases, we have a curve with a minimum for the coals : Ro = 1.4 (good coking coal).

The same is true for the texture (figure 13). With electron microscopy, the ratio of lamella (LMO > 0.2 μ m) is the maximum for an Ro of approximately 1.4. The less sensitive optical microscope shows a minimum of isotropic texture for this rank of coal.

This suggests, on the one hand, that the rank determines the essential characteristics of the coke and that it will be possible to modify the quality of the coke by changing the constituents of the blend and, on the other hand, that these characteristics can be inter-correlated.

We have thus studied a coal blend for finding out the effect of an increased content of low rank coal having a low coking value on the characteristics of the coke (figure 14). The blending of two good coking coals

having similar ranks respects the law of additives, the incorporation of low-fusible (low rank) coal is translated as a positive interaction for the coking coal.

It is also possible to modify the characteristics of the coke by changing the carbonization conditions (figure 15). In this way, resistance to abrasion after gasification improves for increasing carbonization rates. Moreover, the gasification threshold temperature increases with the degree of coke cooking.

It seems, then, that the physical-chemical characteristics of the coke can be changed according to the composition of the coal blend and the carbonization. The textures study allows us to follow the parameters linked to rank more precisely.

V. - COKE CHARACTERISTICS CONNECTED TO REACTIVITY

1. Coke gasification

In the blast furnace, the coke gasification reaction cannot be considered independently from the iron oxide reduction which takes place simultaneously ; the kinetics of the evolution of these two reactions are directly linked. The result, on the average, for the quantity of coke gasified by CO₂ is approximately 25 %, this value being fixed by the availability of the oxygen in the iron oxides.

The global kinetics of the reaction is the consequence of the competition between a chemical reaction and diffusion in the pores : according to the nature of the limiting phenomena, i.e. according to the temperature of the reaction (for a given coke), the gasification reaction can bring about radically different consequences for the porous structure and therefore, for the mechanical strength.

Two extreme cases are illustrated in figure 16. Here we can see the change in the local gasification rate and the local mechanical strength (at the scale of a piece of coke) for a global gasification value of 25 % for the chemical reaction and for the diffusion. The mechanical strength is calculated using a model developed by IRSID which takes the porous structure into account; it is expressed in relation to the initial strength of the non-gasified coke.

In the chemical regime, the entire piece of coke is gasified at the average rate of 25 % and becomes very brittle at the core. In regards to the diffusion regime, the reaction concerns the periphery which is gasified to more than 90 % and which becomes extremely brittle. On the other hand, the core retains the characteristics of the initial piece. In the conditions of the blast furnace, the simultaneity of gasification and abrasion (due to the descent of the burden) causes superficial abrasion of the coke, producing fines and pieces which have reacted only slightly.

The coke gasified in conditions of diffusion shows values to later stress which are greater than that of coke gasified in chemical conditions.

This analysis points out two important parameters for the behavior of coke because they determine the gasification conditions without having any repercussions on the mechanical characteristics :

- the temperature where this reaction began (temperature at the beginning of gasification),
- the intrinsic reactivity of the different carbon textures (linked to the coal rank).

These values are thus to be measured and the effects of certain disturbing elements such as the alkalines are to be analyzed.

2. Differential reactivity of the textures

When 25 % of the coke mass is gasified in a reactivity test, the proportions of the different textures are modified (figure 17). It seems that the relative contents in inert matter and isotropes decrease and that the mosaic contents increase. It can thus be deduced that the inerts and isotropes have gasification rates which are greater than the mosaics.

By taking the bibliography as a reference, we can express the participation of the texture at the speed of gasification by the following equation :

$$V_{\text{Tex}} = 1.72 \% \text{ In} + 1.33 \% \text{ Iso} + 1 \% \text{ Fib} + 0.98 \% \text{ Mos.}$$

3. The influence of ash composition

The accelerating effect of the alkalines on the gasification is well known. Especially sensitive in the blast furnace where they cause fragmentation of the coke, the alkalines have not, nevertheless, been correlated very clearly with the reaction speed.

Various authors have, on the other hand, proposed correlations in function of the basicity of the ashes ; we will cite the following equation proposed by (1) as an example :

$$\text{Bas.} = \% \text{ of ashes} \times \frac{\text{Fe O} + \text{CaO} + \text{MgO} + \text{Na O} + \text{K O}}{\frac{\text{Al O}}{2} + \frac{\text{SiO}}{2}}$$

By combining this equation and the one in function of the texture, we have been able to establish and then experimentally check the following relation which gives the rate of gasification :

$$V = 0.26 V_{\text{Tex}} + 0.15 V_{\text{Bas.}} + 0.24$$

4. Influence of coke degradation in the blast furnace

The differential reactivity of the textures is translated directly as a strong reactivity of cokes rich in inerts and isotropes. The preferential gasification of these textures on the industrial scale has been verified.

This differential reactivity will also cause mechanical behavior which is different after gasification, according to whether the coke is texturally heterogeneous or homogeneous (figure 18). Homogeneous gasification of the material should render the coke less fragile. This postulate will have to be checked.

VI - CONCLUSION

This study underlines the importance of textural characterisation of the blast furnace cokes. A witness of the mean rank of the coal blend, of its homogeneity, the conditions of carbonization, the texture is a parameter which determines the reactivity. Given its influence on hot mechanical behavior, the texture contributes to the permeability of the blast furnace and therefore to its good working order.

(1) Fujita, Hijiriyama, Nishida - Gasification of optical textures of metallurgical coke - Fuel - 1983 Vol. 62 P. 875-879.



SONDAGE TUYERE HF2 SOLMER
TUYERE PROBING BF 2 SOLMER

CARACTERISTIQUES DE LA MACHINE
PROBE CHARACTERISTICS

LONGUEUR LENGTH : 19m
POIDS WEIGHT : 11 tonnes
ENTRAINEMENT DRIVE : 2 *moteurs hydrauliques*
hydraulic motors
GESTION MOUVEMENT : *automate programmable*
MOVEMENT CONTROL programmable logic controller
ALIGNEMENT / AXE BUSILLON : *lunette de visee et laser*
ALIGNMENT / BLOW PIPE AXIS Rifle-scope and laser

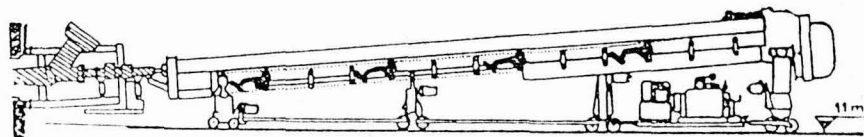



FIGURE 1

	SONDAGE TUYERE HF2	SOLMERS
CAROTTAGES HF A L'ARRET CORE BORINGS DURING BF STOPPAGE		
DIAMETRE SONDE PROBE DIAMETER	: 300 mm	
EFFORT MAXIMAL MAXIMUM FORCE	: 30 tonnes	
INTRODUCTION MAXIMALE MAXIMUM DEPTH	: 6 m/ blindage 6 m/ shell	
MESURES : - effort d'introduction MEASURES	Pushing force	
	- déplacement sonde	Probe positions
	- Localisation des echantillons	Sample positions

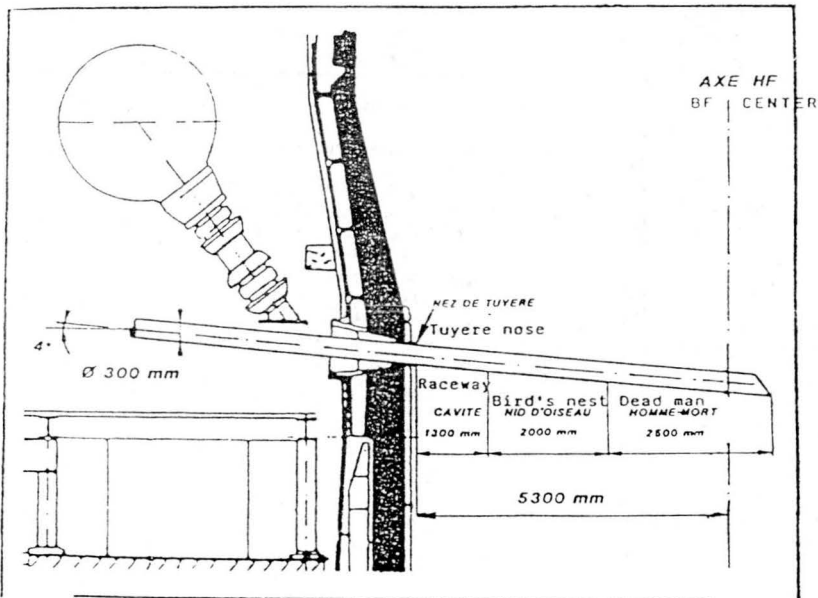


FIGURE 2



	SONDAGE TUYERE HF2									
<p align="center">SONDAGE EN MARCHÉ PROBINGS DURING BF OPERATION</p>										
<table> <tr> <td>DIAMETRE SONDE PROBE DIAMETER</td> <td>: 90 mm</td> </tr> <tr> <td>REFROIDISSEMENT COOLING</td> <td>: eau (65 m /h) Water</td> </tr> <tr> <td>EFFORT MAXIMAL MAXIMUM FORCE</td> <td>: 10 tonnes</td> </tr> <tr> <td>INTRODUCTION DEPTH</td> <td>: 6 m/nez de tuyere 6 m/ tuyere nose</td> </tr> </table>			DIAMETRE SONDE PROBE DIAMETER	: 90 mm	REFROIDISSEMENT COOLING	: eau (65 m /h) Water	EFFORT MAXIMAL MAXIMUM FORCE	: 10 tonnes	INTRODUCTION DEPTH	: 6 m/nez de tuyere 6 m/ tuyere nose
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EFFORT MAXIMAL MAXIMUM FORCE	: 10 tonnes									
INTRODUCTION DEPTH	: 6 m/nez de tuyere 6 m/ tuyere nose									
MESURES MEASURES	<ul style="list-style-type: none"> - effort d'introduction Pushing force - prélèvement et analyse de gaz Gas sampling and analysis - température par pyromètre optique Temperature by means of optical pyrometer - dépot de matières radio-actives Depositing of radioactive materials - pour tracage des liquides to the tracking of liquids - prélèvement de liquides sampling of liquids 									

FIGURE 3

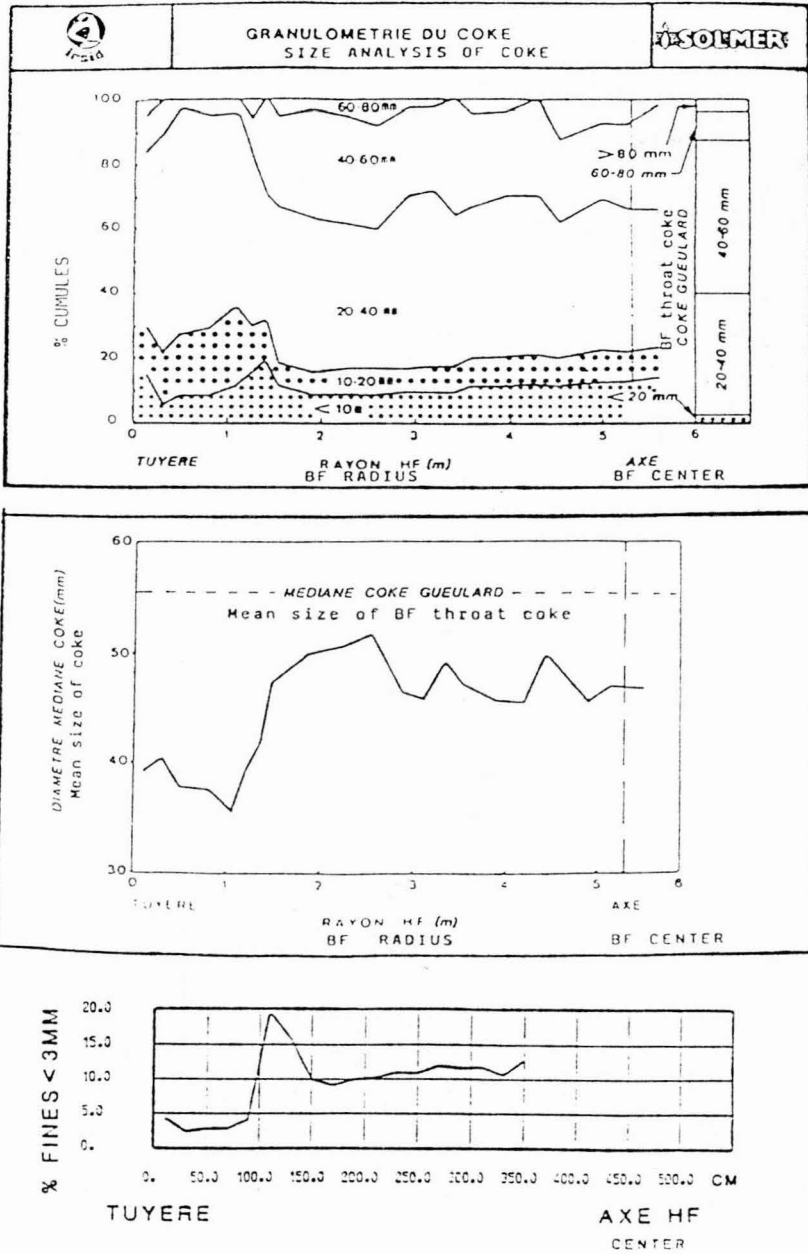
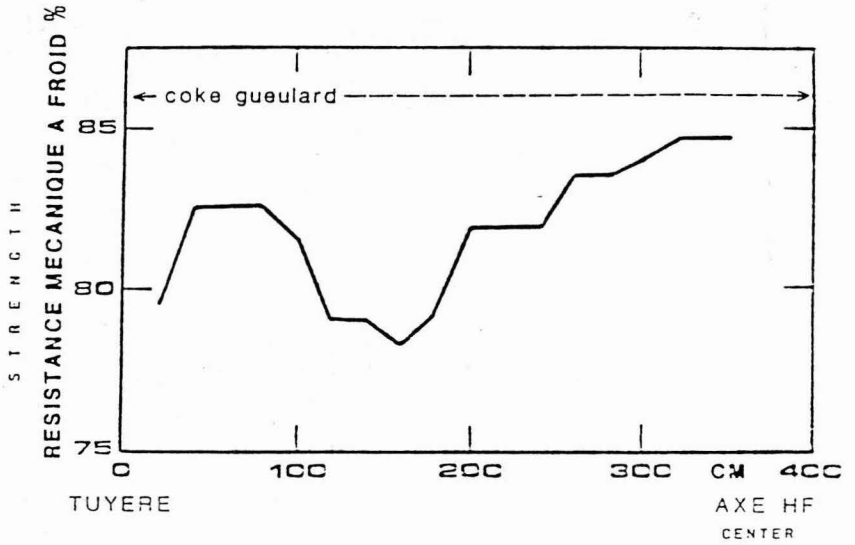


FIGURE 4



EVOLUTION DE LA RESISTANCE MECANIQUE DU COKE PRELEVE
EN FONCTION DU RAYON (CAROTTAGE SOLMER-IRSID)
($\varphi < 3$ mm APRES 500 TOURS TAMBOUR) (FLECHE : COKE ENFOURNE)

CHANGE OF COKE STRENGTH ALONG THE RADIUS
(SOLMER-IRSID TUYERE PROBING) ($\varphi < 3$ mm AFTER 500 REVOLUTIONS IN DRUM)
(ARROW : FEED COKE)

FIGURE 5

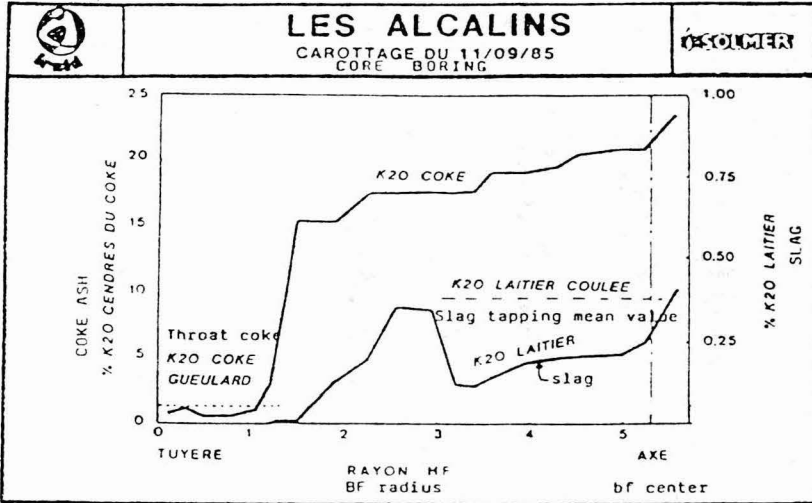


FIGURE 6

EVOLUTION ENTRE GUEULARD ET TUYERES DE LA POROSITE DU COKE
POUR DIFFERENTS CHARBONS D'ORIGINE. POROSITE MESUREE
PAR ANALYSE D'IMAGE

CHANGE OF COKE POROSITY BETWEEN STOCK LINE AND TUYERE LEVEL
AS A FUNCTION OF THE NATURE OF COAL BLEND. (POROSITY FROM IMAGE ANALYSIS)

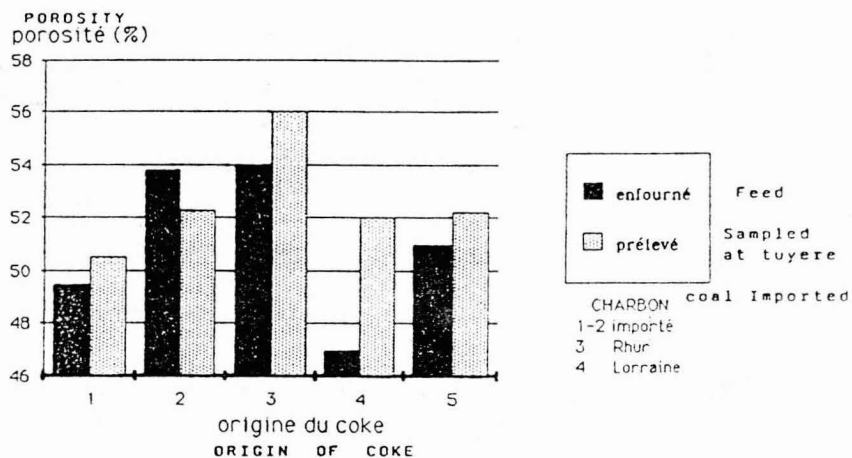


FIGURE 7

EVOLUTION DU COKE DANS
EVOLUTION OF COKE PROPERTIES
LE HAUT FOURNEAU
IN THE BLAST FURNACE

GRANULOMETRIE

SIZE DISTRIBUTION

- Taille moyenne ↓ ↓
Main size
- % Fines < 3 mm. ↑ ↑ (cavité)

RESISTANCE MECANIQUE

STRENGTH

Dégradation faible
Little Degradation

CONSTITUTION CHIMIQUE

CHEMISTRY

- Cendres ↑ ↑ (pollution)
Ash content
- % K₂O ↑ ↑

RESEAU POREUX

POROUS NETWORK

Porosité ± constante
Bulk porosity : constant



FIGURE 8

CPM

GASIFICATION CONDITIONS**CONDITIONS DE GAZEIFICATION****CPM / IRSID**

- 620 → 1200°C + PALIER
- 80%N₂- 10%H₂- 20%CO - 10%CO₂
- Q = 33 l / mn
- 400 g (19 - 21 mm)

Duration = Variable

DUREE = VARIABLE**TAUX DE GAZEIFICATION = CT = -25%**

Gasification degree

→ **VITESSE A 1200°C (g / mn)**

Rate at 1200°C

→ **TEMPERATURE DE SEUIL**

Threshold temperature

JAPON

- 1100°C
- CO₂
- Q = 5 l / mn
- 200 g (19 - 21 mm)

DUREE CT = 2 H**TAUX DE GAZEIFICATION = VARIABLE**→ **% GAZEIFIE CRI ou RI**

x Gasified

MECHANICAL TESTING**TESTS MECANIKUES****- TAMBOUR**

Drum

500 TOURS / 10 MINUTES

Revolutions

⇒ **% < 3 mm / BRUT**⇒ **% < 3 mm APRES GAZ****- CYLINDRE**

Idrum

600 TOURS / 20 ou 30 MINUTES

Revolutions

% > 9,52 mm = CSR ou RS!

FIGURE 9

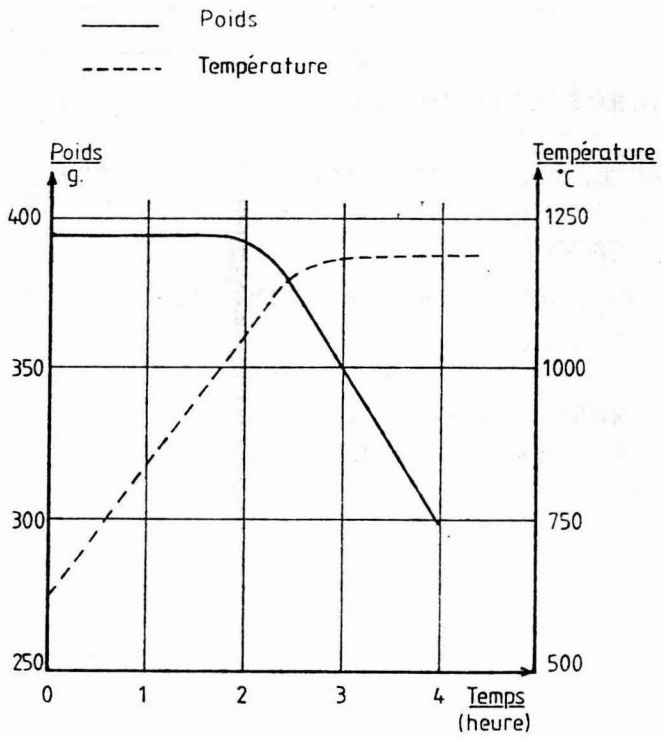


FIGURE 10

TEMPERATURE AND WEIGHT LOSS CURVES AS
GIVEN BY THE CPM-IRSID REACTIVITY TEST

LES DIFFERENTS NIVEAUX D'OBSERVATION DU COKE
THE DIFFERENT LEVELS OF ANALYSIS FOR COKE OBSERVATION

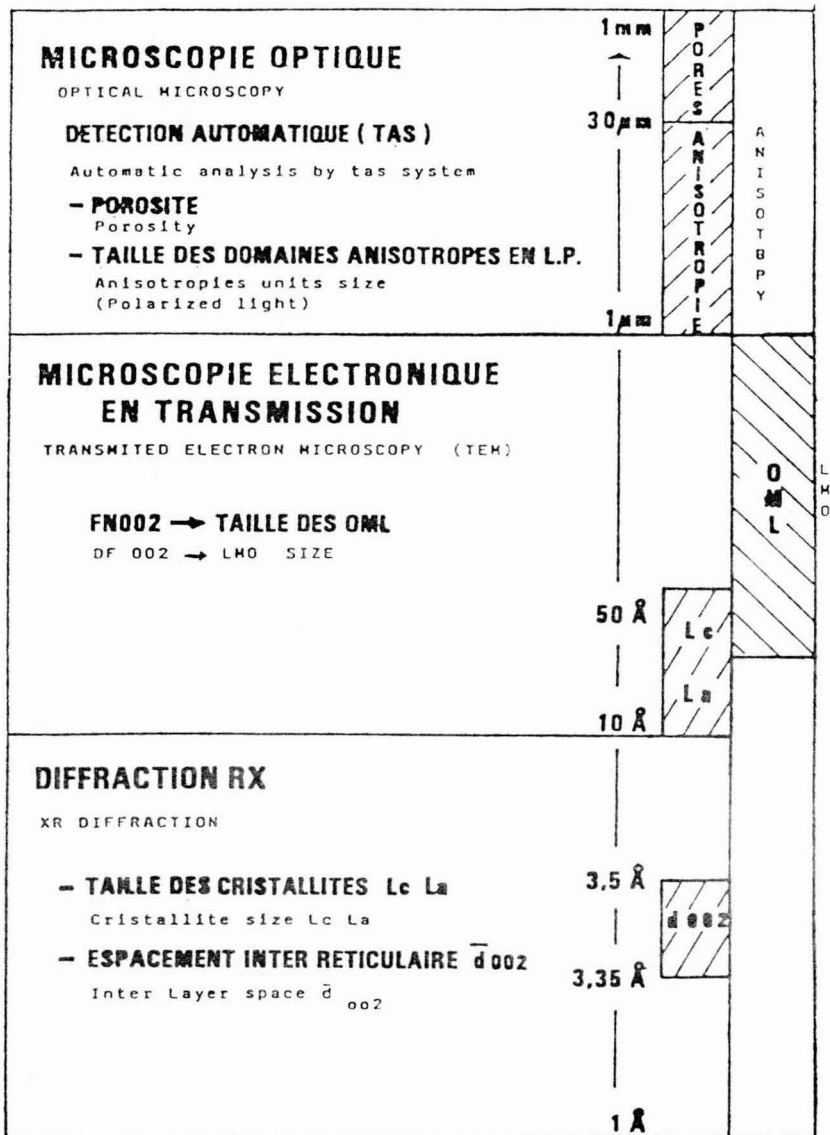
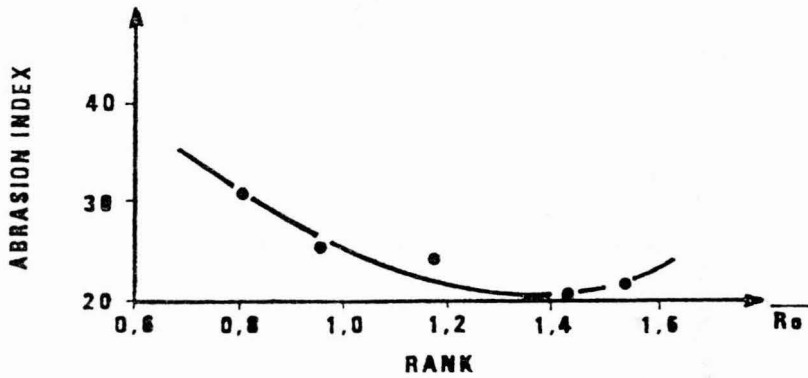
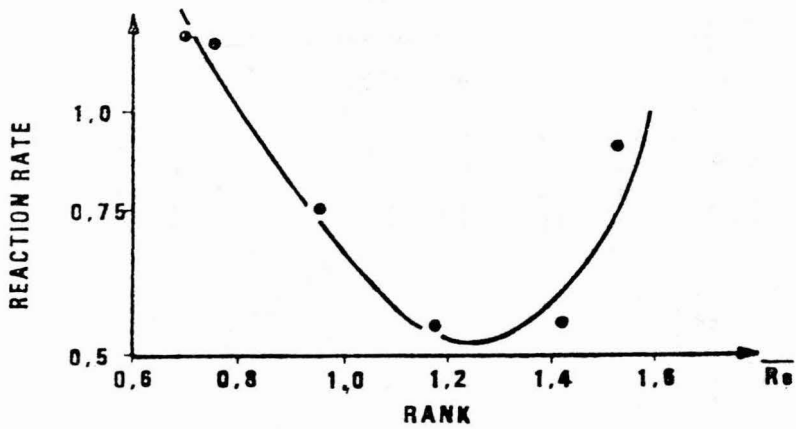


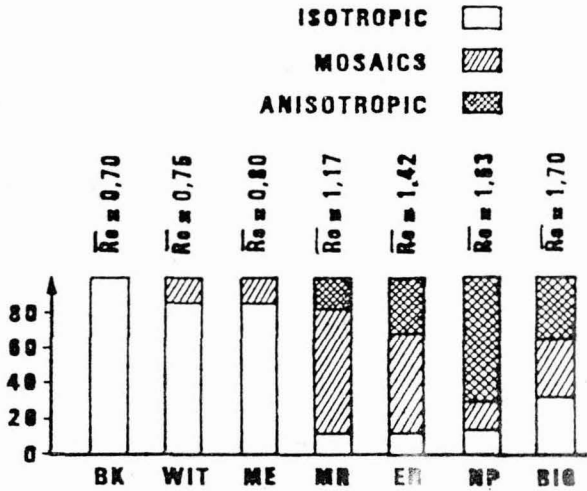
FIGURE 11



EFFET DU RANG DU CHARBON SUR LES PROPRIETES DU COKE
EN COURS DE GAZEIFICATION

EFFECT OF COAL RANK ON POST REACTION PROPERTIES OF COKE

FIGURE 12



EFFET DU RANG DU CHARBON SUR LA REPARTITION DES TEXTURES
 DE LA PHASE CARBONE (MESURE AUTOMATISEE DES TEXTURES
 PAR ANALYSE D'IMAGE AU TAS)

EFFECT OF COAL RANK ON COKE CARBON TEXTURES DISTRIBUTION
 (MEASURED BY AUTOMATIC IMAGE ANALYSIS PROCEDURES)

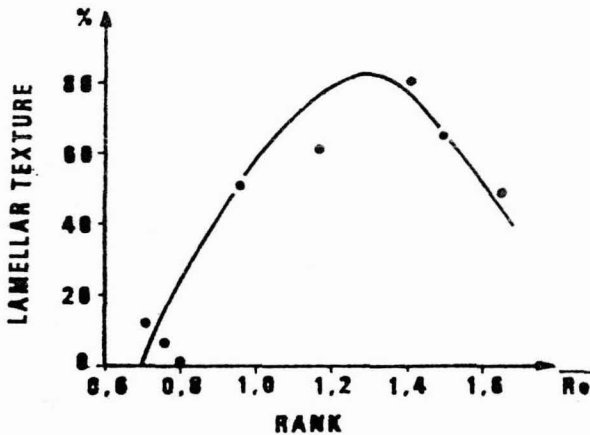
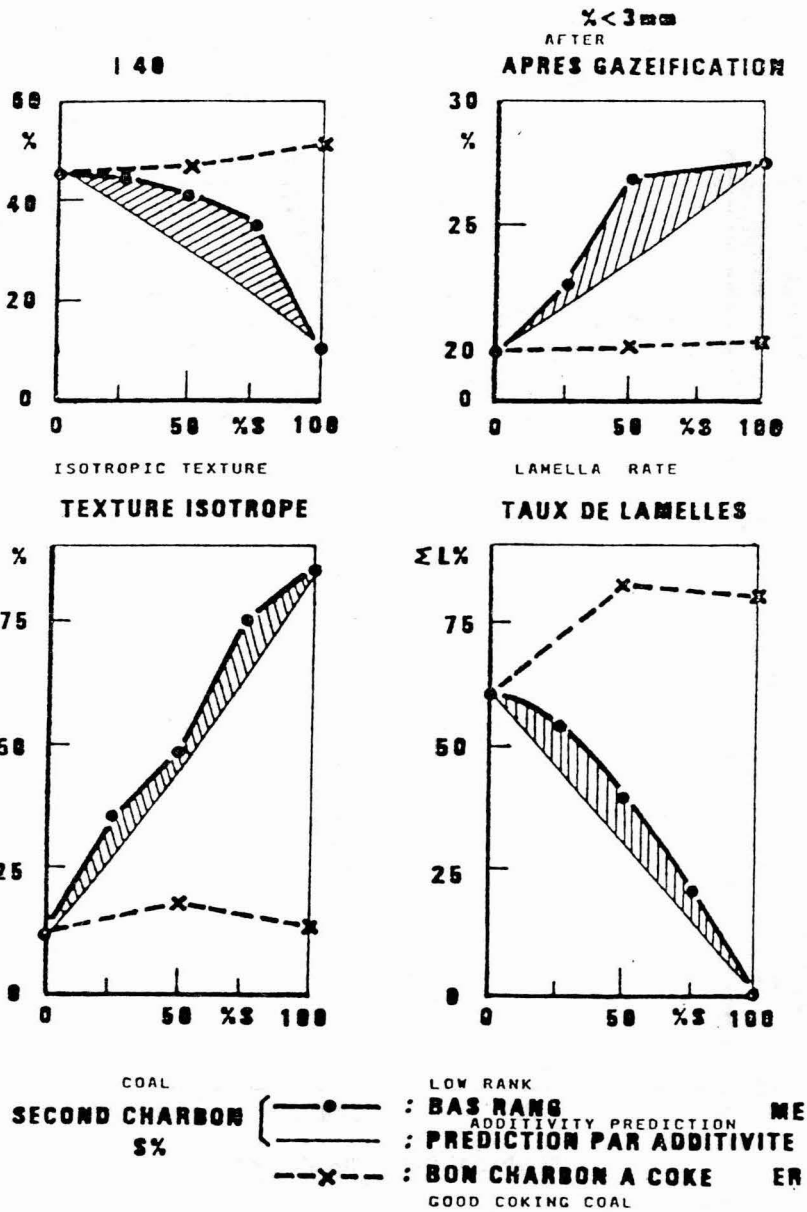


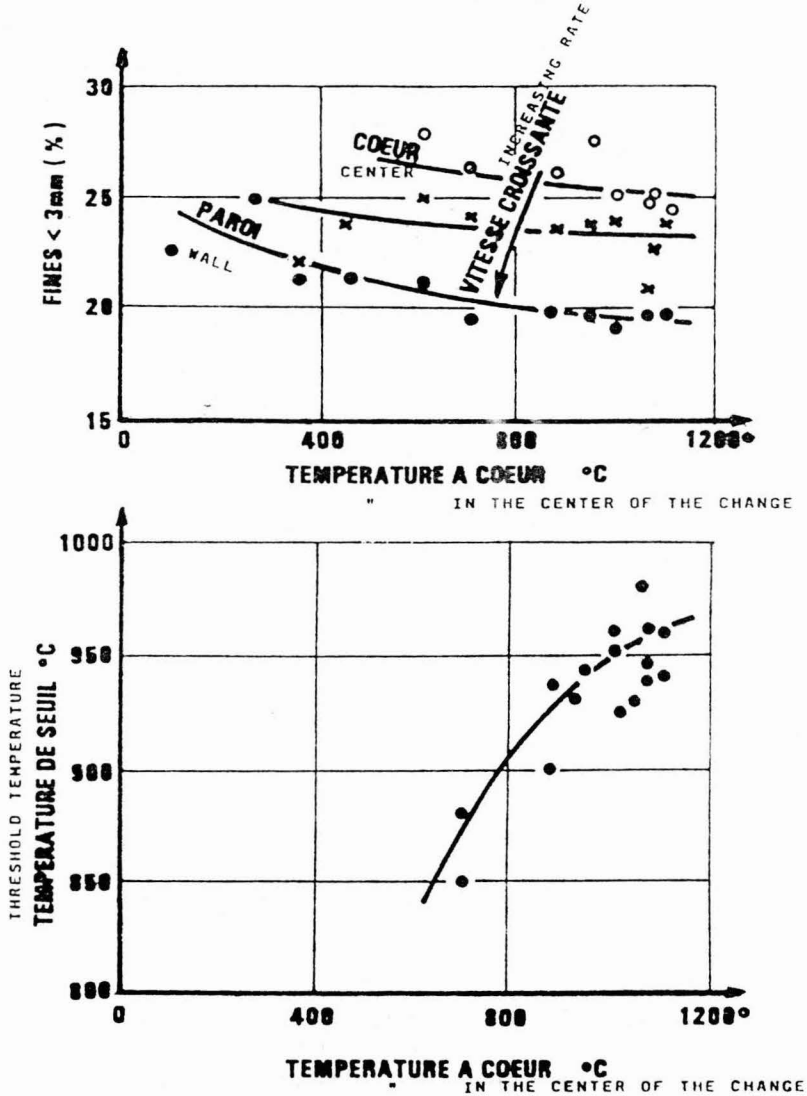
FIGURE 13



INTERACTIONS

FIGURE 14

EFFECT OF COKING CONDITIONS ON COKE POST REACTION PROPERTIES
 (FINAL COKING TEMPERATURE, LOCATION IN THE OVEN)

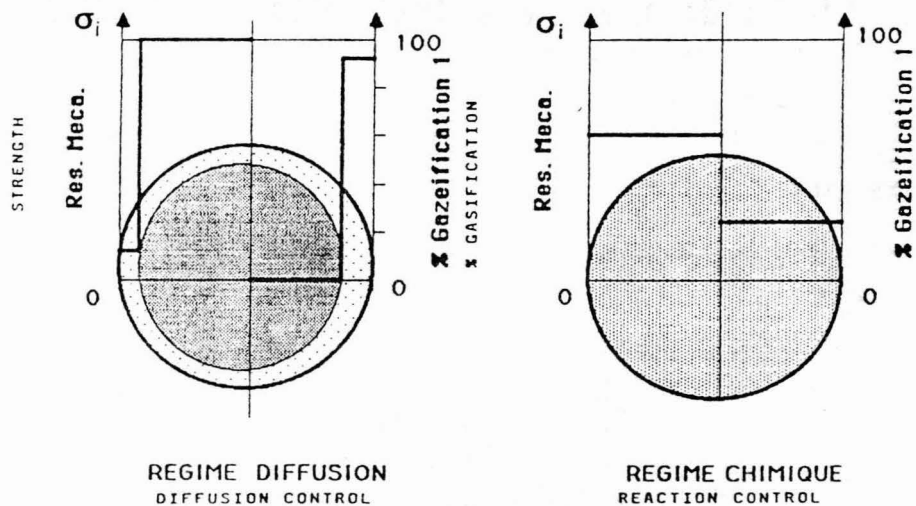


EFFET DES CONDITIONS DE CARBONISATION

(TEMPERATURE A COEUR EN FIN DE CUISSON ET POSITION DANS LE FOUR)

SUR LES PROPRIETES DU COKE APRES GAZEIFICATION

FIGURE 15



GAZEIFICATION

GASIFICATION MECHANISMS

EFFET DU REGIME DE GAZEIFICATION DU COKE SUR LA RESISTANCE MECANIQUE

APRES GAZEIFICATION (TAUX MOYEN DE GAZEIFICATION : 25 %)

(σ_+ : RESISTANCE MECANIQUE INITIALE)

SIMULATION OF THE EFFECTS OF GASIFICATION MECHANISMS

ON COKE STRENGTH AFTER REACTION (AVERAGE GASIFICATION DEGREE : 25 %)

σ_+ : INITIAL COKE STRENGTH

FIGURE 16

GAZEIFICATION DIFFERENTIELLE DES TEXTURES

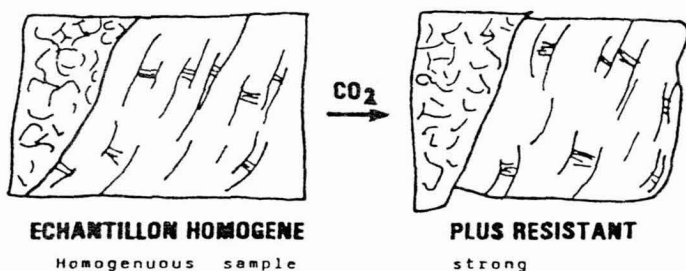
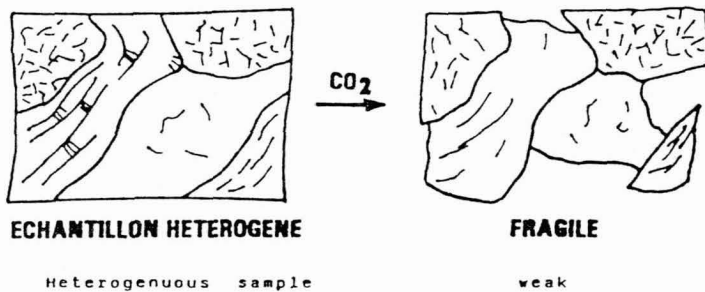
DIFFERENTIAL GASIFICATION OF TEXTURES



$$V_{\text{TEX}} = 1.72 \% I_n + 1.33 \% I_{so} + \% F_i + 0.98 \% M_{os}$$

FIGURE 17

IMPORTANCE PREVISIBLE DE LA REPARTITION DES TEXTURES:
DE LA PHASE CARBONE DU COKE, A L'EHELLE DU MORCEAU,
SUR SES PROPRIETES MECANQUES APRES GAZEIFICATION



EFFECT OF CARBON TEXTURES DISTRIBUTION
WITHIN COKE LUMP ON POST REACTION STRENGTH

FIGURE 18

