ISSN 2176-5480

22nd International Congress of Mechanical Engineering (COBEM 2013)
November 3-7, 2013, Ribeirão Preto, SP, Brazil

ANALYSIS OF ZIRCONIUM DIOXIDE (ZrO_2) SENSORS FOR MEASUREMENT OF OXYGEN CONCENTRATION IN THE EXHAUST GASES FOR AUTOMOTIVE AND INDUSTRIAL APPLICATIONS

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Abstract. *Sensors are critical elements for combustion control and combustion monitoring. Our goal is to improve the performance of the system for example by reducing the levels of pollutant emissions or by smoothing the pattern factor at the combustor exhaust. This research aims to evaluate the progress in sensors in terms of the quality and time of response during operation. The zirconium dioxide (ZrO_2) sensors are a low cost alternative to the measurement of oxygen concentration in the exhaust gases in automotive applications. The methodology applied consists of analyzing the signal of three sensors mounted together in same exhaust pipe, a precise control required to operate this system can only be provided by the use of a Lambda sensor which must be installed upstream of the catalytic converter. The engine is configured by factory to operate with ethanol and compressed natural gas (CNG). State observation and performance estimation are central issues in combustion control. The sensors system needed in this context must feature adequate precision and response, and at the same time cope with the hostile environment prevailing in combustion process. In the respect the main obstacles to the development of combustion control are practical aspects related to: Sensor integrity, reliability, durability and lifetime.*

Keywords: *Zirconium; oxygen sensor; lambda sensor*

1. INTRODUCTION

Today, environmental protection is becoming increasingly important. This fact also applies equally in the field of automotive engineering. Such terms as “greenhouse effect”, “toxic emissions”, “ozone”, and “acid rain” are everyday keywords from the environmental protection vocabulary. The protection of our environment is a matter of concern which is of equal importance not only for politicians, but also for engineers and vehicle operators. Even though the motor vehicle is responsible for only a relatively low proportion of total pollutant emissions, for years now a systematic reduction of the gasoline engine’s emissions has been taking place. These reductions went hand in hand with successive tightening of the emissions limits by the lawmakers in the various countries. All pollutant reduction measures are aimed at achieving a minimum of emissions, while at the same time ensuring minimum fuel consumption, high mileages, and excellent drivability (Bosch, R., 1999).

Society has benefited greatly, over the past century, from advances that have come about in the energy, transportation, communication and medical fields. More recently, society has become increasingly concerned with the unintended consequences of these advances, including global warming, pollution of air and water, and destruction of the ozone layer and forests. Many of these destructive forces can be tied to increased emissions from power plants, home and factory heating units, and vehicles, which derive nearly 90% of their energy from fossil-fuel combustion processes. Add to this various often toxic and/or combustible gaseous and liquid products generated at chemical and materials processing plants, and the need to insure security at airports and other public sites, and it becomes obvious that the means for tracking and controlling such emissions or chemical analytes are required. This article concerns itself, therefore, with gas sensors, reviewing their principles of operation, the progress that has been ongoing in refining their operation and the trends defining where progress is likely to take us in the future (Kim *et al*, 2013).

The Lambda sensor is a device which can be used for automotive applications to control the air–fuel ratio in order to reduce emissions and fuel consumption at internal combustion engines of Otto and Diesel cycles. It is installed in the exhaust gas system and has the purpose of monitoring the concentration of residual oxygen present in gases produced by the engine. Another name for this device is the oxygen sensor from the exhaust gases. The oxygen sensors are very used in industry, medicine and science, but the most common use is in the automotive sector. The reduction in the emission of pollutants generated by the large number of vehicles with internal combustion engines that circulate daily through the streets and roads is of very high importance. Governments, through specific legislation, encourage advances in engine technology, as well as in the improvement of its management, following to gradually reduce the emission levels of pollutants, mainly the gases generated by the combustion of fuels, in order to preserve the quality life and environmental conservation. The Lambda sensor is a vital component in the technology of treatment of the exhaust gases and widely used by vehicle manufacturers for the reduction of emissions by internal combustion engines. In Brazil, recently, the National Environment Council - CONAMA created the Control Program Air Pollution from Motor Vehicles: PROCONVE (cars) and PROMOT (motorcycles) setting deadlines, emission ceilings and establishing technological requirements for motor vehicles, domestic and imported. The Brazilian federal decree 7819, published on 03 October 2012, or Auto-Innovate "aims to support technology development, innovation, safety, environmental protection, energy efficiency and quality of vehicles and auto parts. In this scenario, the Lambda sensor is an essential component in the technology of treatment of the exhausts gases, widely used for the manufacturers of automobiles for the control and the reduction of the emissions of the engines of internal combustion.

In addition to using the oxygen sensor to control the quality of combustion (fuel-air mixture ratio) to reduce emissions / fuel consumption, a three-way catalytic converter system (TWC) is applied. Together they have the ability to treat the three main harmful gases generated during combustion: carbon monoxide (CO), nitrogen oxide (NOx) and hydrocarbons (HC), in gases not pollutants: carbon dioxide (CO_2), water (H_2O) and nitrogen (N_2), improving too considerably consumption and thermal efficiency of the engine.

Several authors cite aspects of the chemical composition, performance within the electrical conductivity (EMF), mechanical strength, response to different potentials of O_2, ability to withstand aggressive environments, among others. Within the application itself, data collection and analysis of the performance of sensors in a real work situation, can be more explored.

The goal of this research is to examine the response of the sensors using liquid fuel (ethanol), and gas (CNG), analyzing the signals generated by them in specific operating conditions and control, noting their response while operating in controlled laboratory.

2. REVIEW OF LITERATURE

Oxygen sensors used in automotive exhaust emission control systems are dominating the applications of solid-state gas sensors in the world. Their triumph began in 1976 after the announcement of stringent emission regulations in California and the demonstration that controlling the air–fuel ratio with an oxygen sensor in conjunction with a three-way catalyst can significantly reduce these emissions (Zechnall, *et al,* 1973; Bosch, 1999). Since this time, the number of oxygen sensors in vehicles is continuously increasing. Another contribution to this enormous growth was the additional requirement of the Californian Air Resource Board (CARB) to monitor all emission relevant components (On Board Diagnosis - OBD), making a second oxygen sensor necessary downstream of the catalyst. Over the last 25 years, the worldwide production level has reached several hundred million parts (Riegel and Neumann, 2002).

There are, in principle, many ways to detect chemical species in the environment. Most commonly, the sensing device takes the form of a chemical to electrical transducer. Classically, this would be in the form of electrochemical cells, operating either in the potentiometric or amperometric mode. Indeed, the sensors installed in tens of millions of new automobiles per year, for the purpose of monitoring the oxygen partial pressure, pO_2, of the exhaust gas, are potentiometric devices utilizing the oxygen ion solid electrolyte, yttria-stabilized zirconia (YSZ). An electromotive force (EMF) is generated across the electrolyte due to the gradient in pO_2 between that in the exhaust manifold and the air reference. This oxygen activity gradient tends to drive oxygen ions by diffusion from the high to the low pO_2 side. (Kim *et al*, 2013).

Sensitivity is the primary property that comes to mind when discussing sensors. This follows from the fact that certain chemical species, even at ppm or lower levels, can be toxic to humans, contribute to corrosion of critical components (e.g. nuclear reactors) and/or poison catalysts essential in emissions control or to the chemicals industry. This brings into play another key sensor property, selectivity, which reflects the often-enormous challenge of selectively detecting small numbers of a specified molecule suspended in a sea of other chemical species, e.g. the surrounding atmosphere. Another important sensor parameter is speed of response. For example, an automotive exhaust sensor must respond within the order of 10 ms to a change in gas composition in order to enable feedback control of the air-to-fuel ratio needed for proper operation of the catalytic convertor. The last of the four key properties is stability, without which reliable sensor readings become impossible. This latter property is becoming more challenging to achieve, as we increasingly require sensors to operate under harsh temperature and environmental conditions. The analysis of the four S's - sensitivity, selectivity, speed and stability - is thus essential in any discussion of chemical sensor development.

For many years, high sensitivity/selectivity sensor systems were limited to the laboratory. More recently, the trend has been away from such large stand-alone analytical chemistry systems (mass spectrometers, chromatographs, IR spectrometers, etc.) that lack portability, require skilled operators and are costly, towards miniature devices, often embedded as part of a sensor array. These lower-cost devices are portable, draw considerably reduced power, and when integrated with appropriate software, provide a level of selectivity impossible with single-sensor-based devices. These advances have been made possible by leveraging corresponding advances in microelectronic and micro-electromechanical (MEMS) processing. At the same time, it must be remembered that while silicon-based chips normally operate at or near room temperature, wrapped in packaging designed to isolate the device from the environment, chemical sensors, on the other hand, commonly operate at elevated temperatures to accelerate kinetic processes and in often harsh chemical environments. This has required the integration of materials not common in the microelectronics field and the modification of the substrates and metallization capable of operating under such conditions. These efforts have been aided by the need for high-power (electric vehicles, power grid controls) and high-temperature electronics (automotive and jet engine controls) where wide band gap materials such as SiC and AlGaN have been introduced and continue to be refined (Kim, *et al*, 2013).

Influenced by common spark plug designs, the conventional first generation zirconia oxygen sensor body had a conical thimble shape. Without high temperature co-firing techniques being developed, the electrodes are applied by thin film techniques after firing, as well as a porous protective layer using flame or plasma spraying methods. This ceramic element is assembled in a stainless steel housing protecting the ceramic against mechanical and thermal impacts. A variety of protection tubes provides the necessary protection of the active part of the ceramic element being exposed to the exhaust gas. The geometry of the openings of the protection tubes also determines the dynamic behavior of the sensor (Riegel, *et al*, 2002).

For the first 15 years, the development was mainly characterized by various efforts to improve its robustness and accuracy in the rough automotive exhaust environment (Gruber and Wiedenmann, 1980; Ueda, *et al* 1994).

The only heat source to achieve the minimum working temperature of approximately 350 jC was the gas temperature itself and it could take a long time to heat up the sensor. To overcome this disadvantage and also to reduce the high temperature variation, the first major improvement during the early 1980s was the introduction of a ceramic heating element (5 to 20 W) inserted into the thimble ceramic as a separate component (Wiedenamnn, *et al*, 1984).

To improve the oxygen ion conductivity, which is decisive for the activation temperature, the low cost Ca-stabilized zirconia has been replaced by the more expensive Y_2O_3 stabilization (Dueker, et al, 1975). Because of problems with the fully (>8 mol%) yttria-stabilized zirconia (FSZ) in respect to mechanical strength and thermal shock resistance, a different kind, the so called ‘‘partially stabilized zirconia’’ (PSZ) with stabilization grade in the range of 4–5 mol% yttria, was chosen by the oxygen sensor industry Table 1. The slightly lower oxygen ion conductivity in comparison to FSZ is overrated by an excellent mechanical strength improvement (Liu and Chen, 1991). However, the complex phase composition of PSZ and the ceramic microstructure needs to be precisely controlled by careful control of the whole manufacturing process (raw material powders, amount of flux, milling/grain size and sintering temperature) to avoid any decomposition or degradation of the mechanical strength. This problem is well known and caused problems in the early vehicle applications in low temperature cycling exhaust gases of 20 to 400 jC (Tsubakino, *et al*, 1991).

Table 1. Stabilization ratio and phase composition of current production zirconia ceramics

Manufacturer		Yttria stabilization (wt. %)	ZrO_2-phase composition (wt. %)		
			Monoclinic	Tetragonal	Cubic
Bosch	Thimble type	9	25	<10	main
NTK	Thimble type	7	~ 0	55	40
Denso	Thimble type	10	23	< 10	main
Delphi	Thimble type	10	20	< 10	main

Potentiometric oxygen sensors are based on the physical behavior of galvanic cells, in which electricity is produced as a result of the spontaneous reaction occurring inside them. In the sensor, a reference partial pressure ref PO_2 is on one side of the electrolyte and an unknown one, PO_2, to be determined is on the other side. An Electromotive Voltage (EMV) in volts, E, is generated between the electrodes by the tendency of oxygen ions within the electrolyte to drift from the high to low oxygen partial pressure side.

The generated EMV between the two electrodes follows the Nerst's equation Eq. (1) (Young et al, 1979).

$$E = \frac{RT}{4F} \ln\left[\frac{P_{O_2}(exhaust)}{P_{O_2}(air)}\right] \tag{1}$$

Were:
R is the universal gas constant;
T is the operating temperature;
F is the Faraday's constant;
$\mathbf{PO_2}$ (exhaust) is the partial pressure of oxygen in the electrode-electrolyte interface of the outer electrode and;
$\mathbf{PO_2}$ (air) is the partial pressure of oxygen in the ambient air (reference gas).

Solid-state oxygen sensors operate on the principle of solid oxide electrolysis often referred to as oxygen pumping. Yttria-stabilized zirconia (YSZ), with porous platinum Pt. electrodes is commonly used for oxygen sensors. Oxygen ion vacancies in the crystal structure of zirconia created by the yttria dopant give rise to selective oxygen ion conductivity.

Application of a potential across the heated YSZ electrolyte is believed to break down molecular oxygen to atomic oxygen that subsequently ionizes to O_2 at one of many three-phase boundaries Fig. 1 on the cathode side. The electric field then drives the ion through the crystal lattice to the anode, where it recombines to form molecular oxygen (Vaniman, 1995).

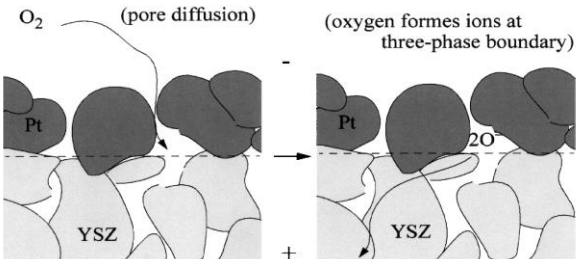

Figure 1. Three-phase boundary between electrode, electrolyte and gas phase. (Sridhar and Blanchard, 1999)

3. MATERIALS E METHODS

For the accomplishment of the experimental assays, an engine produced for FIAT S/A, with exclusivity for the Brazilian market, mounted especially was used so that it functions of independent form to the vehicle Fig. 2 and 3, incorporating all the necessary systems for the perfect performance, thus allowing the accomplishment of tests of controlled form, as well as, allowing the maintenance of the parameters to be analyzed, objectifying the accomplishment of the assays white of this research. Performance of engine, Table 2.

Table 2. Performance of engine - FIRE 1.4 TETRAFUEL

Engine Layout	In line 4
Capacity	1.396 cc
Compression Ratio	10,35:1
Power Output - ABNT (cv/KW)	81/59,6 - 5.500 rpm (ethanol 100%)
	68/50,1 - 5.500 rpm (CNG)*
Torque – ABNT (kgm/Nm)	12,4/121,6 - 2.250 rpm (ethanol 100%)
	10,4/102,2 - 2.250 rpm (CNG)*

*Compressed Natural Gas (CNG).

Figure 2 and 3. Experimental test engine

The selected engine was developed by the manufacturer FIAT, especially to use following fuels: pure gasoline, gasoline with 20% of anhydrous ethanol, ethanol and compressed natural gas (CNG), making possible the evaluation of the sensors of oxygen using combustible gaseous liquid and, thus allowing one better precision, particularly when used the CNG, which had the characteristic of perfect miscibility with the admission air (guaranteed for the use of individual electro injectors of CNG for each cylinder), reducing the variables of imperfection in the air/fuel mixture received per item from the cylinders. For that a perfect combustion occurs, a crucial factor is the maintenance of the relation of the air/fuel mixture inside of the established parameters. Thus the amount of fuel below of the specified one will cause an incomplete combustion of the oxygen, in another extremity the fuel in excess will not be burnt, being banishes from the chamber "in nature" causing the increase of the HC emissions and possible damages to the catalyst of three ways

ISSN 2176-5480

(TWC), amongst others. Being thus, it is necessary that the fuel forms a homogeneous mixture with air, as well as, inside of the correct stoichiometric relation. The correct air/fuel relation (A/F), or stoichiometric mixture, provides to a complete combustion with the consequent decrease emission of gases. The system of electronic management of the engine has immense importance in the correct adjustment of relation A/F, using the signal of the sensor of oxygen for the adjustment of the stoichiometric relation, whichever the fuel used at the moment, making possible a perfect combustion, with consequent reduction of the emissions of gases of exhaustion in function to make possible a correct stoichiometric relation. In Table 3 we have the stoichiometric relation for ethanol and with CNG.

Table 3. stoichiometric values

E100 (ethanol)	AF = 8,5 / 1
CNG (compressed natural gas)	AF = 17,2 / 1

3.1 Methods

To determine the performance of the sensor, it an engine of internal combustion was tested in real situation of work, using, operating with two fuels, individually, the methanol and compressed natural gas (CNG). The sensor target of this research was submitted real the operational conditions, collecting the data instantaneous and its performance, registered and investigated using an equipment of special dedicated diagnosis. The collected data had been processed and analyzed later, searching to deepen the knowledge of the functioning, time of response of the sensor, its durability and capacity to resist the poisoning for diverse chemical elements. For the accomplishment of the assays, dedicated equipment had been used, as described below:

- The Tecnomotor Rasther scan tool, collect sensor signals received by the ECU, necessary adjustments to the operating parameters of the engine.
- Automotive digital multimeter with temperature probes Fig. 4, to collect the temperature of the exhaust gases before the catalyst, for the ideal operating temperature of the oxygen sensor.
- The Minipa MS-1005 automotive portable oscilloscope Fig. 5, to collect additional data signals from sensor.
- The Tecnomotor TM131 exhaust gas analyzer Fig. 6.

Figure 4. Multimeter

Figure 5. Oscilloscope

Figure 6. Gas analyzer

The tests were conducted with the engine operating with liquid fuel (ethanol), and gas (CNG), following the specifications of ABNT NBR ISO 8178-4, was carried out as follows, table 4.

Table 4. Experimental test specifications

Fuel	Sensor	Revolutions (rpm)
Ethanol	A	1000
		2700
CNG	A	1000
		2700

The engine was warmed up to normal operating temperature.

4. RESULTS AND DISCUSSIONS

The ceramic sensors were analyzed by scanning electron microscopy (SEM). The micrographs below show the zirconia surface Fig. 7 and the interface of zirconia and the platinum electrode Fig. 8 (350x) (Authors pictures).

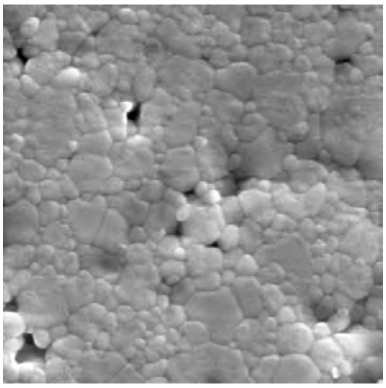

Figure 7

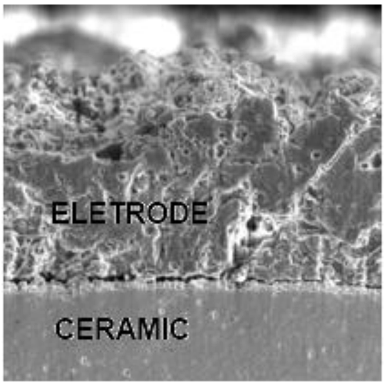

Figure 8

The results of the experimental tests indicate the following signals of the sensor, in milivolts (mV):

- Operating with ethanol

Of table 5 one observes that the mean tension in the sensors when operating with ethanol at 1000 rpm had been of 0,395 mV rpm and 0,537 mV at 2700 rpm.

Table 5. Sensors response - Tension (mV) – Ethanol

Fuel	Sensor	Revolutions (rpm)	V min.	V max.	V average
Ethanol	A	1000	0,073	0,830	0,395
		2700	0,048	0,898	0,537

In figures 9 and 10, we observe the signal of reply of the sensor in the respective rotations.

Sensors response - Signal (mV) – Ethanol

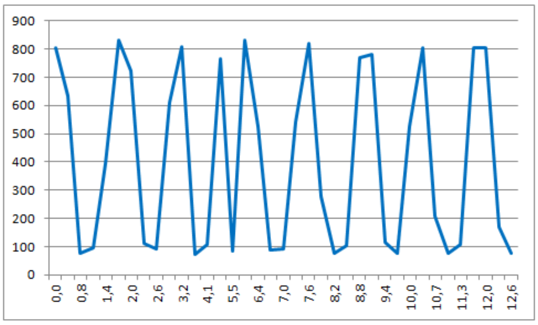

Figure 9. Ø 1000 rpm

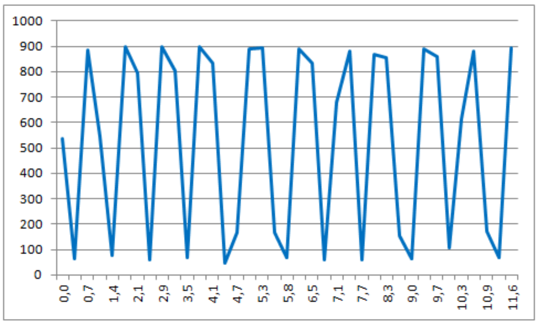

Figure 10. Ø 2700 rpm

- Operating with CNG

Of table 6 one observes that the mean tension in the sensors when operating with CNG at 1000 rpm had been of 0,479 mV rpm and 0,588 mV at 2700 rpm.

Table 6. Sensors Response (mV) – CNG

Fuel	Sensor	Revolutions (rpm)	V min.	V max.	V average
CNG	A	1000	0,068	0,900	0,479
		2700	0,039	0,942	0,588

In figures 11 and 12, we observe the signal of reply of the sensor in the respective rotations.

Sensors Response (mV) – CNG

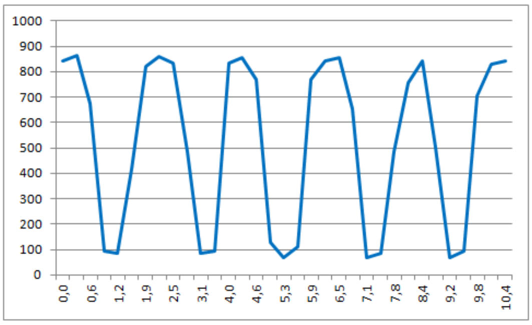

Figure 11. Ø 1000 rpm

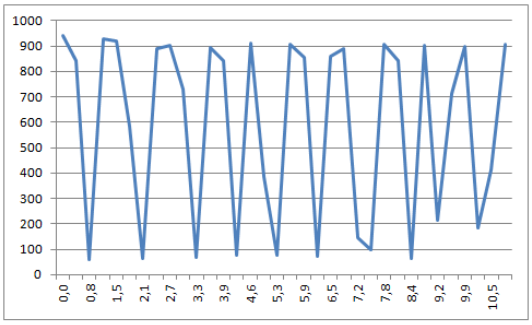

Figure 12. Ø 2700 rpm

As observed in tables and shown figures above, we have: For the 1000 rpm, comparing the results of CNG x ethanol, we verify that the reply of sensors shows a significant difference of 21% (0,395 versus 0,479), already in the rotation of 2700 rpm, carrying through the same comparison we get a 9,5% difference (0,537 x 0588).

In temperatures above of 300ºC the sensing element of zirconia it has the property that causes the transference of ions of oxygen. This movement generates a tension in the order of milivolts. How much bigger it will be to the oxygen concentration enters the gas of escape and the surrounding air (or of reference) in the internal central part of the cone of the sensor, greater the produced tension. The produced tension when the mixture is poor (oxygen excess) is in the order of 0,1 Volts, and in the condition of rich mixture (lack of oxygen) it is of approximately 0,9 Volt. The module in function of the received tension carries through the necessary adjustment of the mixture so that this if keeps the biggest time around the stoichiometric point, 0,5 Volt.

This allows that the management system inside keeps the emissions of the engine of the specified limits. This alteration in the relation air-fuel does not have very to be fast, therefore it can cause imperfections in the engine that would harm the driven by power. To prevent this situation, the ECU contains an integrator that modifies the mixture during one definitive period of time.

A delay also exists enters the formation of the mixture in the collector and the measurement of the oxygen of the exhaust gases. This if must to the cycle of functioning of the engine, the speed of entrance of the mixture, the time for the exhaust gases to reach the sensor, as well as, of the time of response of the sensor. This characteristic time is known as “dead time”, being able to be in the order of as in idling, but only some hundreds of milliseconds in higher rotations. Had to the dead time the mixture it cannot be controlled for an accurate value of $\lambda = 1$. If the integrator is adjusted to allow the rotation of the engine, then lambda is possible to keep the mixture in the window (0,97-1,03), that it is the region in which the catalyst of three ways gets its better efficiency (Denton, 2004).

The table 7 shows the results of the measurement of temperature of the exhaust gases of the region of the sensor:

Table 7. Measurement of temperature - exhaust gases in ºC.

Fuel	Sensor	Rotação (rpm)	ºC
Ethanol	A	1000	400
		2700	770
CNG	A	1000	480
		2700	800

The electronic management system for this engine is the IAW 4SGF denominated Tetrafuel, developed and manufactured by Magneti Marelli brand, using a fuel injector to operate with gaseous fuel (CNG), and with tradit liquid fuel injector. All control engine management, including fuel injection using either liquid or gas is the responsibility of the electronic control module (ECM), based on the received signals, generated by the various sensors distributed throughout the engine. The system is able to self adjust; the electronic unit has the capacity to adjust to small manufacturing tolerances and aging of the engine components.

This control which operates on the basis of the exhaust gas is called closed loop control.

Closed loop: The amount of fuel is determined by the text of residual oxygen in the exhaust gases. In case that it presents oxygen excess the amount of fuel is low, thus being, the electronic management promotes the increase of debit of fuel, in case that it presents low residual oxygen, the electronic management reduces the debit. This method is adopted to guarantee the maximum efficiency of the catalytic converter and optimum fuel consumption.

- Open loop (open circuit): The amount of fuel is determined experimentally in laboratory, where the best dosage is recorded in the memory of the system. This method is adopted to guarantee the maximum performance of the engine in load conditions full and load transitory provisions and rotation.

The experimental measurements had been carried through in the closed-loop condition.

As boarded previously in this work, the factor key for the perfect functioning of the sensor is the operating temperature, thus, how much bigger the temperature of the gases of exhaustion and consequently of the point of installation of the sensor, greater the amplitude and the speed of response of the sensor. This can be confirmed, observing it Table 8 when the CNG as combustible was used, that it presents greater temperature when comparative to the found one when ethanol was only utilized.

Table 8. Values of emissions harvested during the accomplishment of the tests.

Fuel	Sensor	Revolutions (rpm)	CO (g/km)	CO_2	HC (g/km)
Ethanol	A	1000	0,438	171,9	0,023
		2700	0,569	189,2	0,008
CNG	A	1000	0,185	153,1	0,070
		2700	0,621	188,9	0,051

5. CONCLUSIONS

Considering the emissions values, the CNG has advantage when compared with ethanol, a gain in the quality of the response of the sensor, operates with lesser variation, as observed. The results, in accordance with the initial research, show that the best miscibility of the CNG with the admission air, a making possible one mixture more homogeneous, thus offering, one better condition for a complete combustion of the air/fuel mixture, as it can be observed in the reading of response of the sensor and profit in the quality of its response.

Another important factor is that with the increase of the rotation of 1000 for 2700 rpm, the temperature in the sensor went up in function of the biggest flow of gases, passing of 400oC for approximately 800oC, what it reduced the time of reply of the sensor. Comparing still the operating temperatures at 2700 in both fuels, are observed that the CNG comparatively presented superior temperatures at the ethanol. The mean stress in the region of sensor using itself CNG superior when was compared with the ethanol in both the rotations.

The strategy of adjustment of the mixture can be verified in the long time with clarity, demonstrated in the graphs of tension x rotation, adopted in the programming of the module, aiming at the improvement of driveability and optimal functioning of the engine. Such strategy is only possible with the response of quality generated for the sensor.

6. ACKNOWLEDGEMENTS

The authors would like to acknowledge the suppots from **CAPES -** Coordenação de Aperfeiçoamento de Pessoal de Nível Superior; **CNPq - C**onselho Nacional de Desenvolvimento Científico e Tecnológico; **FAPESP** - Fundação de Amparo a Pesquisa do Estado de São Paulo; **SENAI -** Serviço Nacional de Aprendizagem Industrial **-** DR São Paulo; **FIAT** Automóveis S/A; **TECNOMOTOR -** Eletrônica do Brasil S/A.

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