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IMPROVEMENT OF SINTERING PROCESS OF PLATINUM LAYERS IN ELECTRICAL FURNACES

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ABSTRACT

With the increased requirements for the price and quality of the components used in automotive industry and also the enhancing criteria for on time deliveries, which are related with keeping the stock of parts in warehouses very low, more and more manufacturing companies of these elements are forced to optimizing measures and fitting-up of high-tech equipment. As a rule, before such investments, very serious economic rationale, detailed risk assessment and long functional tests, followed by customer validations and finally approval must be done. Each of those steps guarantees the quality and the expedience of the project. The technology of production of electro-resistive temperature sensors for the automotive industry, based on thin film platinum layers, uses many thermal processes on different stages of their elaboration as a main method for stabilization of each next applied micro layer. To their running are demanded great and diverse requirements, which enforce a high level of the equipment and the technologies.

The present work aims to the improvement of two of the sintering processes of platinum paste, applied directly to the ceramic base of the sensor element, using identical thermal regime. Their implementation is realized in terms of changing the existing older type of furnaces with new advanced version of these aggregates with perfected design of the operating chamber and improved control of the temperature in it.

An evaluation of the thermal work of standard furnace for the production and also of the new advanced type is made. Both results are compared. It is analyzed the influence of the sintering process on the treated platinum layers and the ceramic substrate. All received data are confronted with their relevant ones at using the standard units and conditions. The obtained results allow the conclusion, that the replacement of the equipment is reasoned and it not only improves characteristics and product quality, but also reduces variations in the performance of the process. It was analyzed the influence of the sintering process on the platinum layers and the ceramic base. All the obtained data are compared with those corresponding to them using standard conditions and aggregates.

Keywords: electrical furnaces, sintering, sensors, temperature control, energy efficiency.

INTRODUCTION

The high-temperature sensor elements type Pt200 represent a ceramic base with applied platinum and insulating layers in a particular configuration and construction, depending on the specific characteristics of the product and its application. To their manufacturing are brought stringent and diverse requirements necessitating a high level of equipment and technologies [1].

In Fig. 1 is shown a schematic construction of a standard sensor element TS200A [2], where every layer, applied trough screen-printing method, is presented in a separate level above the ceramic base, according to the sequence of application.

The sensing elements are processed on a total number of ten pieces on one ceramic base, and after the completion of the screen-printing operations and treatments, are separated into single positions.

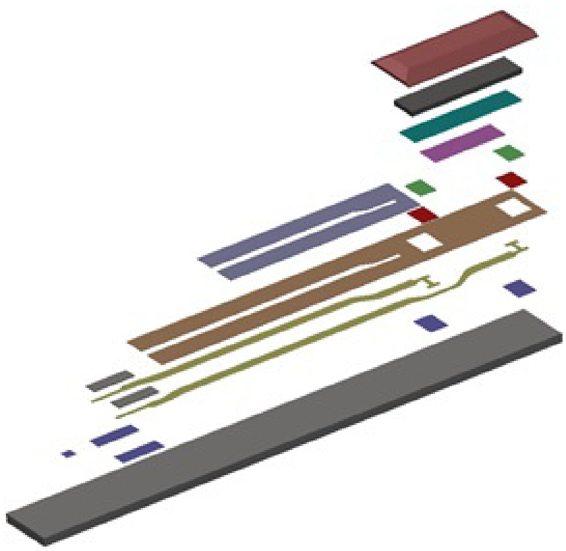

Fig. 1. Construction of a sensor element type TS200A [2].

The main interest in the present study is the firing of the first two layers, processed at the highest temperature, directly contacting with the ceramic substrate, and on which surface all other layers of the sensor element are built.

Fig. 2 shows the temperature curve of the common chamber process used for sintering of the first and the second layer of platinum pastes in conventional furnaces type Rohde KE155 [3].

Every section (step) of this temperature process plays a role in the formation of qualitative and secure layer with precisely defined parameters and thickness. The maximum and most critical process temperature is 1340 + 10/-5°C for 35 min. It is proven, that this narrow range should be followed strictly, as temperatures below the lower limit lead to poor adhesion of the layer to ceramics and such upper limit cause distortion of the substrate and layer thickness below the specified tolerance.

In previous our work [4] the possibilities for optimization of the production capacity of electrical chamber furnace, using optimal layout of the workspace and production are presented. Despite the encouraging results obtained, this approach has limits to improve the energy and environmental efficiency of the production process. Therefore, in order to increase its parameters, more radical steps have to be undertaken, related with the respective investment and at the base on their appropriateness, technical-economic calculations will be imposed [5].

The used furnaces so far Rohde KE155, are bizonal chamber units with heaters made from material Kanthal APM [6]. They use contactor control power, have maximum operation temperature of 1350°C and nominal power consumption 32 kW.

With the increase of the production, the number of heat treatments also increases, leading to more frequent preventive repairs of heaters, refractories and contactors. The heaters have a maximum operating temperature of 1250°C and melting point 1500°C [6]. Their usage at values above the maximum specified by the manufacturer, drastically reduce their lifetime. In nominal process temperature of 1340°C (which is very close to the maximum one of the furnace 1350°C), inevitably increase the aging of the heaters and wear of the refractory lining. It is estimated that maintenance of a furnace for a period of three years is equal to its cost as new, but scrapped production in case of accident during

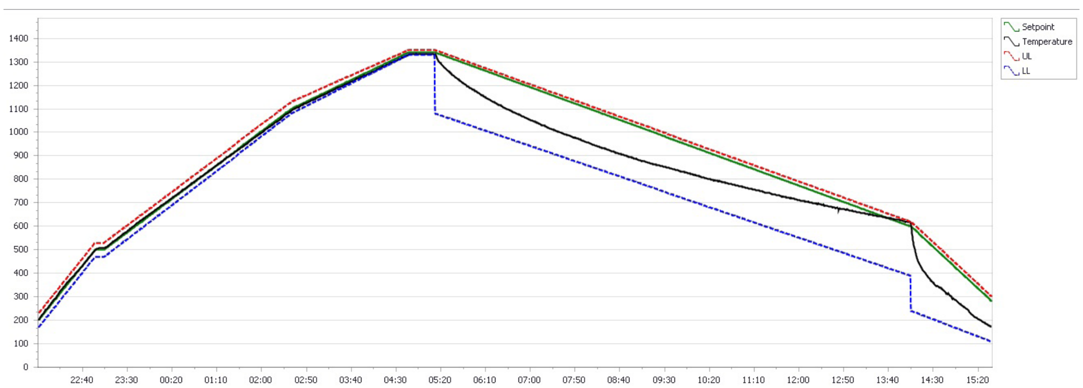

Fig. 2. Temperature curve at sintering of the first and second platinum paste layers in a standard furnace type Rohde KE155 [3].

Table 1.

Furnace type	Rohde KE155	Nabertherm HTC160/16S
Useful volume, l	155	155
Nominal power, kW	32	21
Maximum work temperature, ºC	1350	1550
Heaters type	Kanthal APM	Kanthal Globar (SiC)
Process control type	contactors	Thyristors
Number of zones	2	1
Heaters lifetime, years	≈ 1	≈ 4
Time for one working cycle, h	23	19

firing is at even higher value [7].

The mentioned facts above led to a decision to be searched a new type of high-temperature chamber furnace, which is more resistant to wear and tear, and at the same time, if possible, to have higher efficiency and better control of the process.

Having in mind the functional problems of the used aggregates, it was concluded, that the choices of furnaces of higher grade are these types, which use a ceramic heaters – SiC [8] or $MoSi_2$ [9]. They have longer operation at 1350°C compared with heaters based of metal or metal alloys [10] and they allow the usage of precise thyristor control [11].

The present work aims to the improvement of two of the sintering processes of platinum paste, applied directly to the ceramic base of the sensor element, using identical thermal regime. Their implementation is realized in terms of changing the existing older type of furnaces with new advanced version of these aggregates with perfected design of the operating chamber and improved control of the temperature in it.

EXPERIMENTAL

After careful study of the high-temperature furnace's market [3, 12 - 14] and having in mind the technology requirements, it was decided the aggregates for this process to be replaced with one-zone furnaces Nabertherm HTC160/16S [15]. They are with almost cubic chamber, ceramic heaters type Kanthal Globar® (SiC) [16], thyristor control of power supply, maximum temperature 1550ºC and nominal power consumption 21 kW.

Comparison between the basic technical characteristics of the old and the new furnaces, is presented on Table 1. Typical for the furnaces Nabertherm HTC160/16S is the usage of light, porous refractory material for the construction, which significantly increases the energy efficiency of the unit, and the speeds of heating and cooling. The symmetrical furnace chamber in combination with thyristor control, suggests better temperature oversight compared to the old version, in which the chamber is divided vertically into two zones with separate contactor control of the power supply.

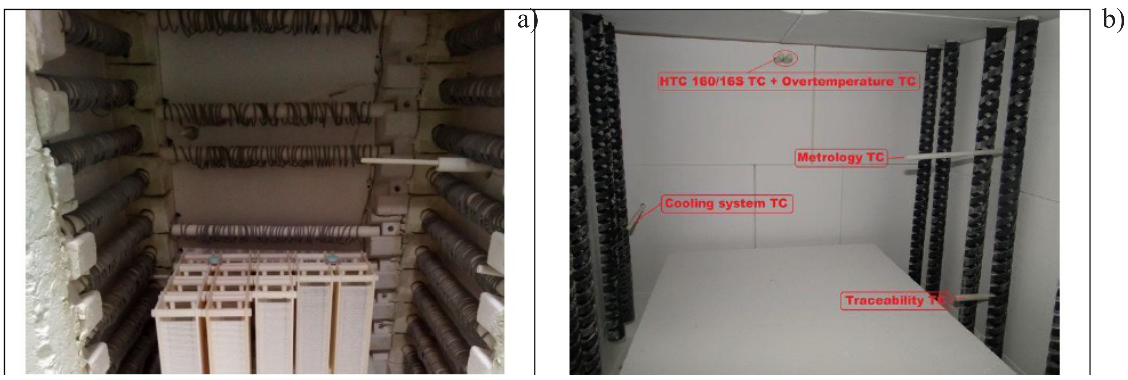

Fig. 3. Common view of the furnace chambers: a) Rohde KE155 with positioned thermocouple for calibration measurement; b) Nabertherm HTC160/16S with thermocouples for process control, recording and calibration measurement.

A thermocouple type S and device for recording and storing data Almemo 2690 [17] are used for independent temperature reading. The sensing tip of the thermocouple is positioned in the volume of the chamber at a distance of approximately 0.350 - 0.4 m from the wall surface and measures the temperature of the furnace atmosphere in the range above the load (Fig. 3).

RESULTS AND DISCUSSION

Thermoprofiles and temperature field in the furnace

The profiles of both furnaces in the part of heating plus the time at the maximum temperature, and accordingly the consumed power for achieving them, are traced by the real output of the firing of the first and the second layer (Fig. 4). For recording the energy released from the heaters at any point in time, a specialized software is used, which reads the data directly from the controllers of the furnaces [18].

Since, as noted above, the new furnaces Nabertherm are cooled down more quickly due to the lightweight construction of the chamber, it was proposed a controlled, linear chilling from the maximum temperature of 1340°C to 1100°C, thus avoiding the stress in the product from the rapid temperature change of the furnace atmosphere in a free cooling mode. To achieve this linear portion upon chilling, the controller of the furnace doses accurately the power of the heaters through thyristor control, which achieves a certain speed of the process.

Fig. 5 shows a comparison between the cooling rates of the old and the new furnaces.

Assessment of process parameters and quality in the new and the old furnaces

During the above described processes data were collected for the distribution of temperature field in the volume of the fired products, acquired through the use of heat-shrinkable rings (thermorings) with a working range of 1130 - 1400°C and accuracy ± 3°C. These gadgets are made of special cements, able to shrink to exact level, depending on the maximum reached temperature. The reading is performed by measuring the current dimension of the ring, using a special micrometer [19]. Data for diameters are compared to the specification provided by the manufacturer of the rings for the

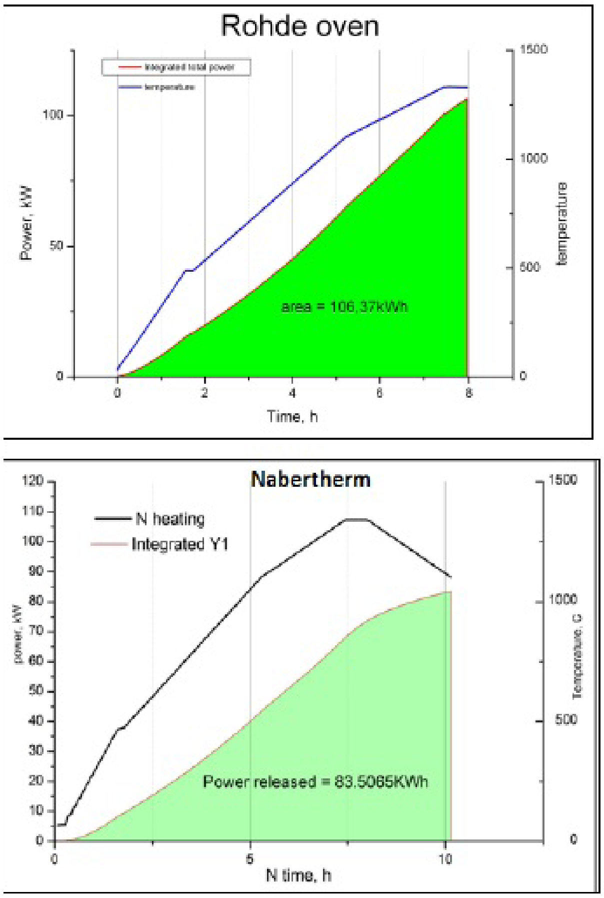

Fig. 4. Thermoprofile of heating and holding at a maximum temperature of the furnace: a) Rohde KE155; b) Nabertherm HTC160/16S, with added linear cooling step to 1100ºC.

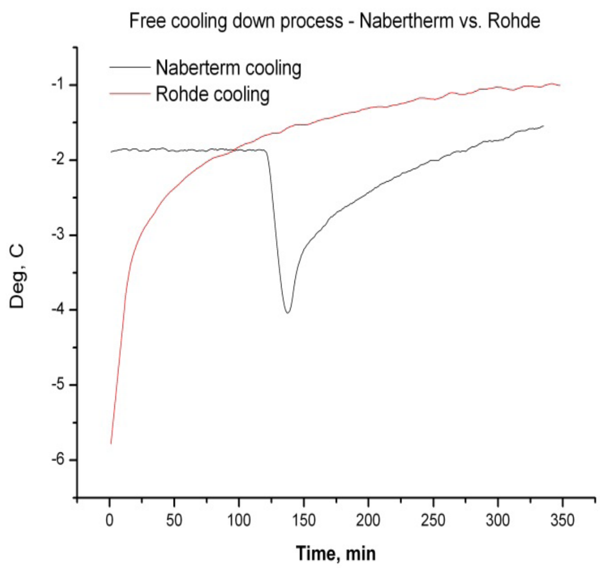

Fig. 5. Comparison between the speeds of cooling in both types of furnaces.

Fig. 6. Common view of shrink ring on a ceramic washer.

batch, where for any value of the diameter of the ring the corresponding temperature is indicated. Calibration of temperature readings takes place in the manufacturer's laboratory. Placed at different points of the chamber, the rings provide information about the local temperature in each of them.

Fig. 6 presents a picture of heat-shrinkable ring on a ceramic plate, which prevents the sticking of the ring on a rough surface. Schemes for arrangement of the rings in both types of sintering for the first and the second layers, are shown in Fig. 7.

As can be seen, for the firing of the first layer 34 ceramic holders are used, placed one over the other horizontally. In them the product (50 ceramic substrates with 10 sensor elements each – a total of 500 pieces) is stored. For the firing of second layer are used up to 22 ceramic holders, which contain in total the same amount of product, but this time they are loaded vertically. This difference is related to the specific technology of production, aimed ceramic substrate to keep as flat as possible, regardless of the high temperatures and its small thickness - below 0.001 m.

At firing of the first layer, the thermorings are arranged by five in 3 levels, while at the firing of the third layer they are in 2 levels, again in 5 pieces.

Schemes for arrangement of the thermorings for both ways of charging are given in Fig. 8.

Comparisons between the acquired information on the distribution of temperature field in both types of furnaces, recorded by the thermorings for different types of arrangements, are presented in Fig. 9.

The results show, that in terms of the temperature field, the new ovens are fully viable and meet the requirements of the process. During the sintering of the second layer a more even temperature distribution is observed, partially of better symmetry of the heating chamber and to some extent of the added linear section at cooling, which increases the average time at above 1300°C and that way improves the homogenization.

The temperature curves of the process are compared for the both types of furnaces in Fig. 10.

Comparing the temperature profiles it may be concluded, that in the part of the heating and holding, the processes in both furnaces are identical. When a problem occurs, due to the more rational disposal of the control thermocouple (in the inner top of the camera), the new furnace would react more adequately and will compensate the missing heat increasing the power to

Fig. 7. Common view of the arrangement in sintering: a) the first platinum layer 34 (horizontal) ceramic holders; b) the second platinum layer 22 (vertical) ceramic holders.

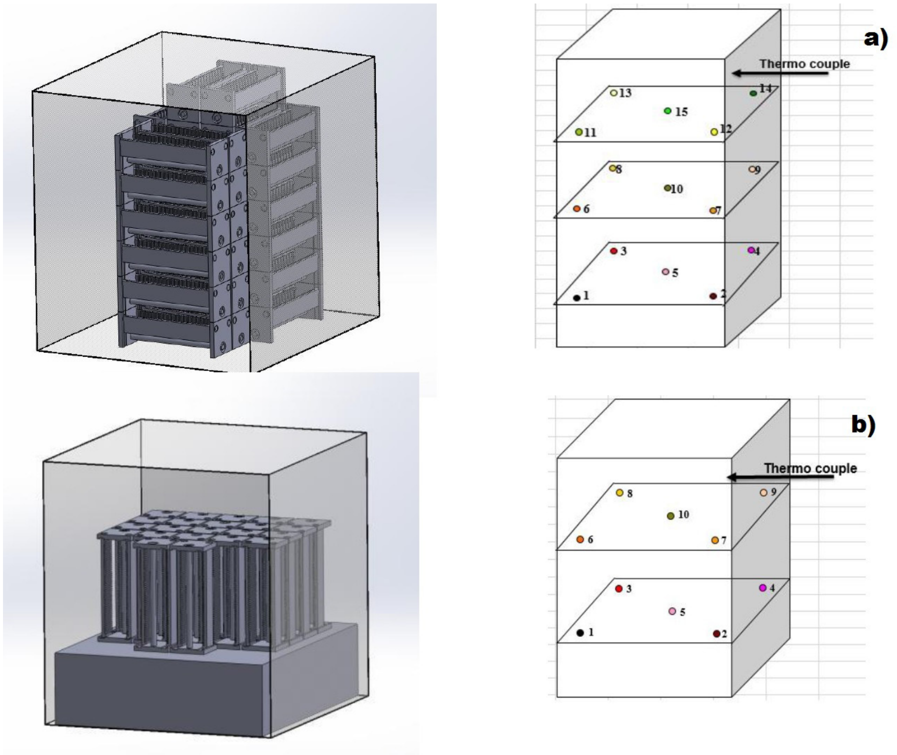

Fig. 8. Scheme of the arrangement of thermorings in production volume for sintering of: a) the first layer; b) the second layer.

the remaining heaters. In the old model furnace the compensation occurs from the zone that is not affected by the problem. That way, the temperature in it arises to values outside the specified tolerances, which leads to large amounts of rejects.

In the cooling down step of the new furnaces the option with a linear segment in the range 1340 - 1100°C is chosen to eliminate the stress in the production from rapid shutdown of the supplied thermal power, which on its part compensates to some extent the heat from the massive refractory wall at the original process in the old type furnaces. Even at this certain delay, the total time for heat treatment is reduced by four hours, that in total for the two layers makes eight hours saved time (one production shift). As can be seen below, this difference does not affect the quality of the product, and even improves it. Despite the extra power delivery during this interval, for one thermal processing are totaly saved about 22.86 kWh of electricity, which based on approximately 150 high-temperature treatments per month, means 3.43

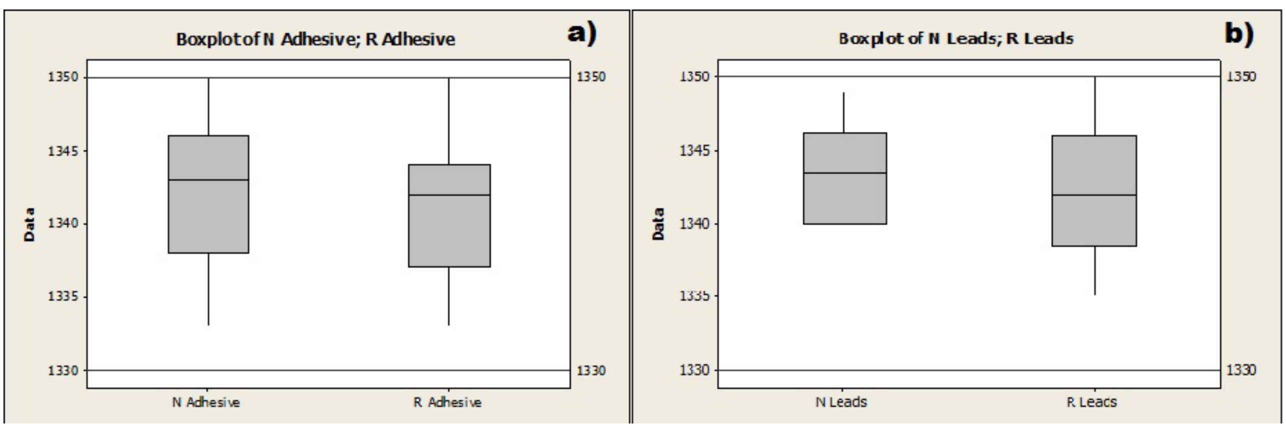

Fig. 9. Temperature distribution in production volume after sintering of: a) the first layer; b) the second layer; N - Nabertherm HTC160/16S; R – Rohde KE155.

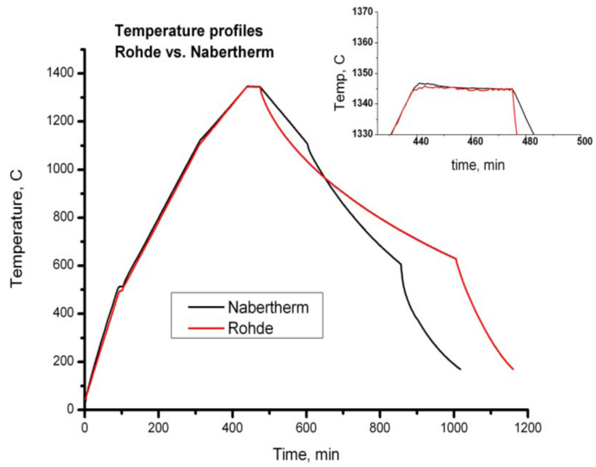

Fig. 10. Comparison between the process temperature curves for furnaces Rohde KE155 and Nabertherm HTC 160/16S.

MWh electricity saving. It increases the energy and environmental efficiency of the review process, which achieves a complex techno-economic effect.

As mentioned, a major problem in the firings of the first and the second layers is the bending of the ceramic substrates, due to the heating and their stay at a temperature of 1340°C. The deviation of flatness of the ceramic substrate is measured after the temperature processes for both types of furnaces (Fig. 11). The sample size based on 50 ceramic substrates is used, since for each of them is measured the maximum deviation of flatness of both diagonals and the bigger value is taken for the purpose of this study.

As can be seen from the comparative graphs, the data distribution for bending between test substrates, processed in the new and old furnaces, is fully comparable between them, and within the working borders $\pm 10^{-4}$ m. After the process of firing of the first layer, the flatness is kept significantly better in the substrates that were thermally treated in furnace Nabertherm HTC160/16S.

Produced in both thermal units test elements are assembled to ready for mounting in car sensors and are put under different functional tests (mechanical and thermal) for proving and comparison of their properties.

One of the most significant tests is so called thermoshock, where the sensor is placed in a combustion chamber with a flow of exhaust gases, directly after the flame of burner. Thereby are simulating the toughest conditions, under which work sensors type TS200A – in the exhaust manifold of the engine. The products of combustion heat the sensor up to 1000°C for a period of 26 s. Immediately after reaching this temperature, an automatic machine puts the sensor into spray atmospheric air and it is cooled to room temperature again for 26 s, which closes one cycle of the test. As a measurer for the quality of the sensor, the number of cycles is counted that it can withstand before the appearance of a functional defect -destruction of the glass insulation coating, mechanical damage in the ceramic base, loss of signal from the sensor, deviation from the precise temperature values, etc.

Comparisons between the results of the thermoshock test of sensors, manufactured in the old and in the new

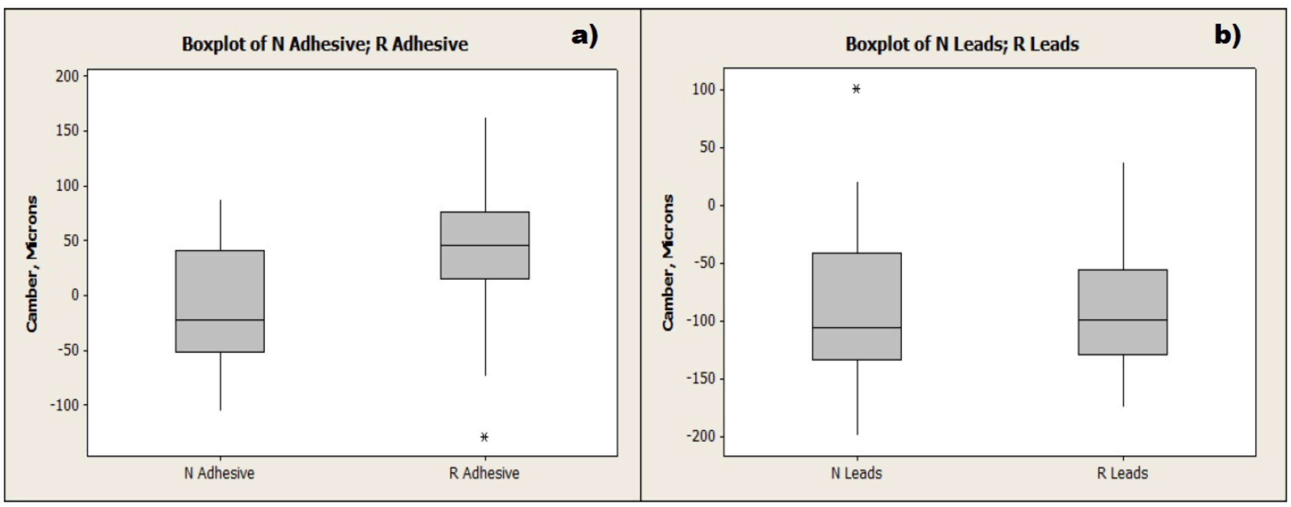

Fig. 11. Comparison between the measured values of deviation of flatness after sintering of: a) the first layer; b) the second layer; N – Nabertherm HTC160/16S; R – Rohde KE155.

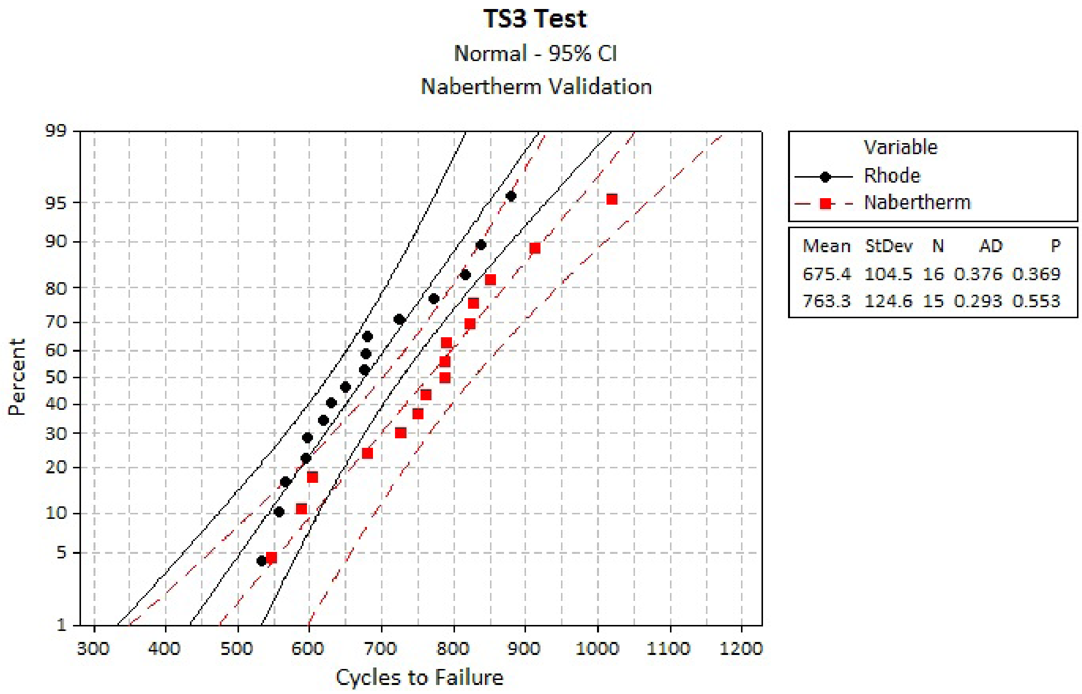

Fig. 12. Results from the thermoshock test.

furnaces, are presented in Fig. 12.

As can be seen, the strength of the elements processed in the new furnaces, expressed as a number of the thermoshock cycles passed, is nearly about 14 % higher than that one of the standard produced in the furnaces Rohde KE155. The distribution of the data results for the quality of the two test groups sensors shows, that there is significant difference in favor of those produced in the new furnaces Nabertherm HTC160/16S.

CONCLUSIONS

From the conducted tests for evaluation of the suitability of the proposed new type furnaces and the specific conclusions made, it follows that the aggregates Nabertherm HTC160/16S have a number of advantages over the prior art Rohde KE155. The most remarkable among them are reducing the production time per unit of output, energy savings and hence improving the energy and environmental efficiency of the whole technological process, lowering the cost of maintenance of equipment, enhancement the operating time, improved control and monitoring of the process as well as a significant increase in the quality of the manufactured sensors, ensuring their flawless operation in the car.

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