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RESEARCH ON MICROWAVE HEATING CONDITIONS OF CORDIERITE CYLINDRICAL SHAPE FOR AFTER TREATMENT APPLICATIONS

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Abstract: *The present paper proposes the realization of some researches on the heating conditions of the cordierite ceramic monolith in unidirectional microwave field for the optimization of the reduction processes by selective catalysis of NOx emissions from non-road transport systems. Previous research has revealed the possibility of rapidly reaching the temperature required to initiate the catalytic reaction for injected power of 1200 W microwaves, cordierite monoliths having different sizes and shapes. Research has also shown that the ceramic support of the catalyst interacts best with microwaves when its shape is cylindrical. The paper aims to optimize microwave heating mechanisms by exposing cylindrical samples with different diameter / heights ratios.*

Key words: *cordierite, microwave heating, selective catalytic reduction, NOx reduction.*

1. INTRODUCTION

Non-road mobile machinery (NRMM) represents an important sector of transport systems taking into consideration its contribution to industry growth. However, the latest decisions related to emissions limits of combustion engines stated in the EU regulation 2016/1628, requires new approaches in the development of new category of low pollution engines as well as in retrofitting the old ones. According to EU reports [1], in 2015 the shipping emissions represented around 13% of the overall EU greenhouse gas emissions from the transport sector. The total greenhouse gas (GHG) emissions from international shipping increased with 31.7% in Europe in 2017 compared cu 1990 [2]. The reduction of NOx emissions as part of the EU effort in reducing pollution has been taken in consideration by developing different technologies funded by major EU projects [3] as well as by academia. Langella [4] has evaluated the environmental impact of maritime traffic in port areas for engines using heavy fuel oil (HFO) for different operating regimes of marine engines. The emissions of NOx for 25% engine load were 17.2 g/kWh much more than accepted emissions (3.50 g/kWh for engines with net power higher than 560 kW) by Stage V of Regulation 2016/1628. The engines tested were MAN DIESEL 14 with 9800 kW. On the other hand, one of the most promising technology that can be developed for reducing the NOx emissions from inland ship combustion engines is selective catalytic reduction (SCR). However, the technology reduces up to 90% of NOx emissions if the reaction temperature is higher than 300^0 C. According to the researches performed by Zahn [5], the primary SCR reaction using V-SCR based on V_2O_5-WO_3-TiO_2 can be initiated for an effective temperature window between 300^0 C and 450^0 C. Culbertson [6] has shown that below 300^0 C the presence of moisture in catalyst will affect the performance of the SCR. The Cold-Start Effect (CSE) of the diesel engines led to higher NOx emissions mainly in port areas where the concentration of the pollutant emissions is high. Their research was focused on introducing of an additional heating source in order to maintain the proper operating temperature of SCR in order to reduce the unwanted transition period from a CSE to nominal operating regime. They introduced a heater element composed from a resistance unit with 1 kW net power in order to increase the temperature of the catalyst even under unfavourable cold start conditions, i.e. at very low exhaust gas temperature. The experiment shown that a system heated for 520 seconds reaches temperature of 170^0 C some 80 seconds earlier than a system without heating. The NOx emissions on the tailpipe decreased with 67% when the heater have been applied. The experiment have been performed on Euro 6 engine.

This paper aims to demonstrate that microwave technology can be used for heating the ceramic core of the SCR and to optimize the heating mechanism in order to avoid unwanted phenomenon like thermal runaway or microwave plasma. The technology for reducing near to zero of the NOx emissions uses a SCR activated with microwaves.

Microwave heating of materials have been widely researched by scientists in order to synthesize new materials with higher properties. Ceramics are the materials with best absorbance properties of microwaves and conversion of the high frequency electrical waves in to heat. According to Guo [7], the microwave heating can be used for rapid synthesis of rutile TiO_2. Being a ceramic, the titan oxide can be fast heated if the microwaves are applied. Basak [8], in his study, uses ceramics as support for microwave heating of other materials. Singh [9] reported how Al_2O_3 can be used as support for different classes of materials that could not be heated with microwaves due to their reflection coefficient. Mangra [9] and Stefan [10] reported in their studies the synthesis of barium ferrites using microwave heating. Regarding the microwave heating of ceramics, Savu [11, 12] and his collaborators reported that one of the most unwanted phenomena is related to thermal runaway of the materials during heating process. The research presented have been oriented to the optimization of the heating process in order to avoid the thermal runaway phenomena. Dietrich and collaborators [13] investigated the interaction between microwaves and ceramic core of SCR. Their researches were focused on the experimental determination of the effective complex dielectric properties of zeolite coatings on cordierite substrates and the approximation of the latter. The research has shown that the dielectric properties of the SCR core can improves the heating mechanism of the microwaves.

Peng [14] has investigated the concept of MW-SCR (microwave – selective catalyst reduction), also. Activated carbon supported ceria and mixed metal oxides MeOx–CeO_2 (Me = Ni, Mn, Fe) were investigated for microwave irradiation-selective catalytic reduction (MW-SCR) of NO in the microwave catalytic reaction mode. NO conversion almost reached 100% for 5% (21% NiOx)-CeO_2/AC at 300^0 C, 5% (21% MeOx)-CeO_2/AC (Me = Mn, Fe) and 5% CeO_2/AC at 350^0 C. MW-SCR of NO by AC primarily produced N_2 and CO_2, and O_2 existence had no influence on the activity of NO removal. These results show that microwave irradiation exhibit the microwave catalytic effect and microwave selective effect. This method of MW-SCR is a viable and promising method for flue-gas such as NO, N_2O, and SO_2 control. The hybrid MW-SCR system was also developed for denitration process.

Beside the conversion of microwaves into heat, the main problem is related to stability of the heating process. The ceramic core of the SCR is obtain from cordierite ($Mg_2Al_4Si_5O_{18}$) and current researches performed by the authors [15] have concluded that the preheating time of ceramic core of SCR is lower in case of microwave heating than other auxilliary thermal sources like resistance heating. However, the thermal runaway occurs and the temperature increases very fast for injected powers higher than 1200 W. In addition, the most stable heating process has been obtained for cylindrical shape of cordierite composite material. Also, other researches proves that cylindrical shapes of the materials are suitable for microwave processing. Navarro [16] developed and validates a numerical method based on a spectral collocation method to predict the temperature of a cylindrical sample heated radially by microwave by solving Maxwell’s equations. Hossan and his collaborators [17] studied the evolution of the thermal field in cylindrical shapes when materials were exposed to microwave. They concluded that the temperature distribution in food cylinders reveals that a slight change in length could cause a significant change in temperature variation within the sample. Dinani [18], Basak [19] reported in his research paper that the cuboid shape in the cylindrical shape in the second place are preferred to be used for microwave heating.

2. MATERIALS AND METHODS

Previous researches [15, 18] performed in microwave heating of cordierite have shown that cylindrical shapes are the most suitable for fast heating in microwave field. However, the results reported that microwave plasma has been initiated above 350^0 C. The samples used in experimental program had different dimensions that can be consulted in the table below. The cordierite samples were obtained by milling from a commercial monolith used in after-treatment application from automotive industry.

Table 1

Dimensions of cordierite samples used in experimental program

Code	d [mm]	h [mm]	Ratio d/h
P1	290	590	0,49
P2	170	500	0,34
P3	200	460	0,43
P4	250	320	0,78
P5	120	330	0,36
P6	100	400	0,25

The determination of the ratio [d/h] is important to predict how the samples will behave in microwave field compared with a commercial cordierite monolith used as ceramic support in SCR applications.

The process heating parameters were established starting from past results obtained. The samples were exposed to 600 W, 1200 W, 1800 W and 2400 W microwave injected power and the heating time has been recorded when the temperatures reached 250^0 C and 350^0 C. Those temperatures have been taken into consideration, as been the temperature interval where the SCR reduces more than 90% of NOx emissions. The microwaves were generated by a 6 kW Muegge Microwave System with power adjustable from 600 W to 6000 W. The system contains a water-cooled magnetron at 3 bar pressure and managed by a three-phase power source cooled with water in open circuit. The heating process is controlled by a matching load impedance auto-tuner Homer driven by a stand-alone software Tristan where the process can be monitorized and tuned. The temperature were sensed with a non-contact temperature sensor (infrared pyrometer) Optris G5H with range between 250^0 C and 1650^0 C. The emissivity of the cordierite has been established experimentally at 0,9243 using an infrared pyrometer and a thermocouple type K with range between 0^0 C and 1300^0 C. The process has been also monitorized with a Logitech 3000 webcam and a process computer where the data were recorded.

3. RESULTS AND DISCUSSIONS

The results obtained are presented in the table below.

Table 2

Heating time of cordierite for different microwave injected powers

P [W]	t_{250} [min]	t_{350} [min]	Ratio d/h
P1			
600	0,16	0,50	0,49
1200	0,29	0,54	
1800	0,16	0,35	
2400	Plasma MW	Plasma MW	
P2			
600	0,23	0,36	0,34
1200	0,15	0,53	
1800	> 6,00	Plasma MW	
2400	Plasma MW	Plasma MW	
P3			
600	1,24	1,46	0,43
1200	4,15	4,31	
1800	Plasma MW	Plasma MW	
2400	Plasma MW	Plasma MW	
P4			
600	1,00	1,38	0,78
1200	4,56	> 6,00	
1800	Plasma MW	Plasma MW	
2400	Plasma MW	Plasma MW	
P5			
600	> 6,00	> 6,00	0,36
1200	> 6,00	> 6,00	
1800	> 6,00	> 6,00	
2400	> 6,00	> 6,00	
P6			
600	3,00	5,11	0,25

1200	0,35	0,45	
1800	Plasma MW	Plasma MW	
2400	Plasma MW	Plasma MW	

where:

- Parameter **P [W]** represents the total injected power of microwaves into the cordierite samples
- Parameter **t_{250} [min]** represents the total heating time until the temperature reaches 250^0 C considered to be the required temperature for initiation of the process related to selective catalytic reduction of NOx emissions
- Parameter **t_{350} [min]** represents the total heating time until the temperature reaches 350^0 C considered to be the optimum temperature for common selective catalytic devices of combustion engines from automotive industry
- ratio d/h – ratio between diameter and height of the ceramic monolith substrate (cordierite) sample

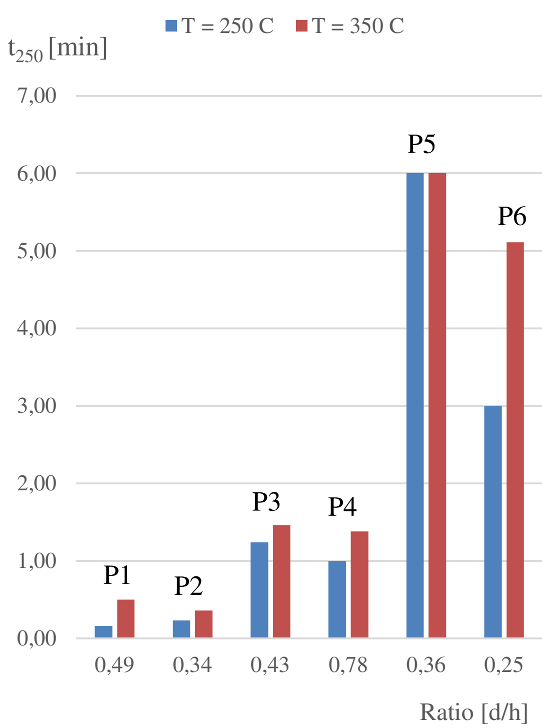

Fig. 1. Total heating time as function of ratio

For the sample P1, having ratio 0,49, was obtained the best heating time for 600 W and 1200 W. The process was quite stable at 1800 W. However, at 1800 W, small arc discharges and local heating occurred at the coupling system between heating chamber and waveguide. The internal temperature of the matching load auto-tuner increased at 42^0 C and the process has been stopped in order to avoid the damage of the auto-tuner.

For the sample P2, having ratio 0,34, the best heating time was obtained also for 600 W and 1200 W. By increasing the microwave injected power, the process became unstable and the microwave plasma was initiated without obtaining the heat inside the samples.

For the sample P3, having ratio 0,43, the heating process was stable for 1200 W. However, initial sample was destroyed by microwave plasma after 1,46 minute due to contact between samples and metallic walls of the heating chamber.

For sample P4, having ratio 0,78, the process was relative stable for low and medium values of the injected power. For 600 W the plasma arc occurred and for 1200 W the temperatures increased at 328^0 C after 5,15 minute. After 5,50 minute the temperature start to decrease due to occurrence micro-discharges between coupling system and waveguide.

For the sample P5, having ratio 0,36, the process was stopped after 6 minutes taking into consideration that the heating time is too long and therefore cannot be applied with success.

For the sample P6, having ratio 0,25, the process was slow for 600 W and very fast for 1200 W. By increasing the power to 1200 W and 1800 W the plasma arc was initiated and the process have been stopped.

In terms of temperature evolution, only 4 samples were taken into consideration due to the stability of the process and occurrence of microwave plasma or thermal runaway. More, sample P5 could not be heated in time limit established for heating process.

The figure below presents the temperature evolution of samples P1, P2, P4 and P6 for 1200 W microwave injected power. The graph shows a smooth evolution of the temperature for P4 which has the ratio 0,78. The other samples presents a very good time for heating, but the thermal field inside the cordierite was not uniform. By using a secondary infrared pyrometer [23, 24], with temperature range for measurement between 0^0 C to 900^0 C, provided by Sensortherm GmbH, different values of the temperatures have been recorded in different points on the surface of the samples.

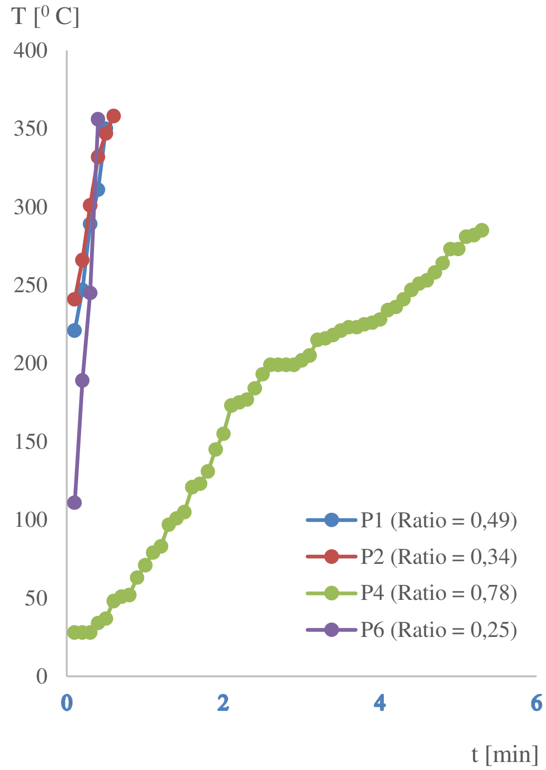

Fig. 2. Temperature evolution in cordierite material

The mathematical model for temperature evolution of the selected samples is presented below [15].

$$T_{SCR} = a \cdot t_{SCR} + b \quad (1)$$

where:

- Parameter $\mathbf{T_{SCR}}$ **[^{0}C]** represents the temperature required by after treatment system for reducing the NOx emissions
- Parameter $\mathbf{t_{SCR}}$ **[min]** represents the total heating time required by after treatment system for reaching the catalytic temperature
- Parameters **a** and **b** are calculated, using a linear approximation of the temperature evolution in time

The process parameters **a**, **b** are presented in table below. Their values depend of shapes, dimensions and position of the cordierite inside the heating chamber. Also, their values depend of power of the microwaves injected in cordierite samples.

Table 3

Parameters a, b for different ratio of the samples at 1200 W microwave injected power

Code	Ratio d/h	a	b
P1	0,49	322,00	187,00
P2	0,34	245,43	221,60
P4	0,78	50,98	33,25
P6	0,25	791,00	27,50

The conclusion was that the microwave heating of the samples with ratio below 0,78 does not be heated uniform in very short time. The mathematical model for all 4 processes counted by experimental research can be expressed in the following equations.

$$T_{SCR} = 322{,}00 \cdot t_{SCR} + 187{,}00 \quad (2)$$

$$T_{SCR} = 245{,}43 \cdot t_{SCR} + 221{,}60 \quad (3)$$

$$T_{SCR} = 50{,}98 \cdot t_{SCR} + 33{,}25 \quad (4)$$

$$T_{SCR} = 791{,}00 \cdot t_{SCR} + 27{,}50 \quad (5)$$

By maintaining the level of injected power to 1200 W, the results obtained have shown the increasing of temperature beyond 350^0 C in some points of the surface, but a large areas remained at temperatures below 250^0 C. From selective catalytic reduction process point of view, these results cannot be taken into consideration knowing that the temperature for activation of catalytic process must be stable and uniform distributed in material's volume.

The optimization of the heating process was elaborated on basis of the results presented below. The samples P1, P2, P4 and P6 presented a stable behavior in microwave field, but in all cases have been reported small arc discharges and high local heat, which can led to the damage of the auto-tuner and microwave generator's antenna. Therefore, the optimization of the heating process consists of:

- Designing a cooling flange between heating chamber and waveguide in order to reduce the internal temperature of matching load auto-tuner.

- Introducing a dielectric ring inside the heating chamber in order to reduce the exposure to the peaks of microwave beam.
- Establishing the optimized position of the three stub tuners in order to reduce level of reflected power from the samples towards microwave generator [21]
- Designing the extension of matching load auto-tuner' waveguide in order to dissipate the heat from the samples and to avoid the local overheating of tuner.
- Designing and producing a thermoplastic double wall with dielectric properties from composite materials [22] in order to reduce the possibility of occurrence of micro plasma discharges between heating chamber and microwave wave guide

The figure below presents the heating chamber after and the coupling system to the waveguide. The main improvements made it were related to extension of waveguide (WR340 type) in order to reduce the influence of the temperature on the matching load auto tuner. The edges of the extended waveguide were welded on the main body of the heating chamber.

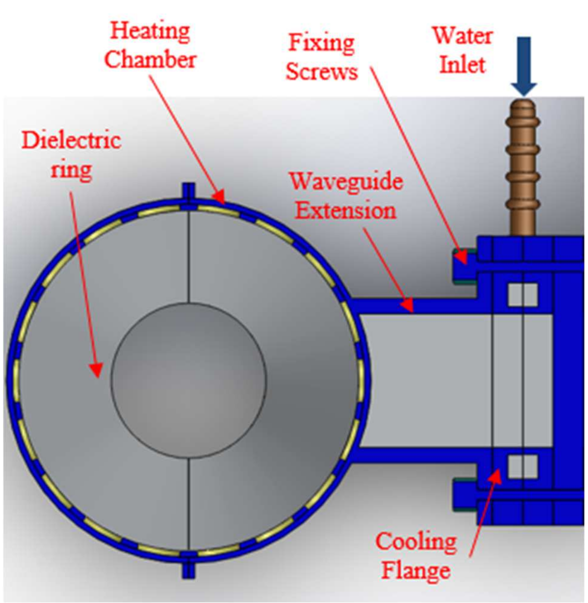

Fig. 3. Heating chamber and cooling flange for high power of microwaves injected in cordierite samples

The connection to the Tristan matching load auto-tuner has been done with screws in order to provide access to the microwave installation. In addition, the cooling flange was placed between Tristan auto tuner and extended waveguide. The inlet water, for cooling the flange, had 3 bar pressure provided by an external circulating pump. The dielectric ring was realized from commercial Al_2O_3 having purity 99,9% and grain size 200 μm pressed at 250 MPa and sintered in microwave field. The dielectric double wall was realized using 3D printing technologies [25, 26]

The overall dimensions of heating chamber corresponds to real size of SCR device used in automotive industry.

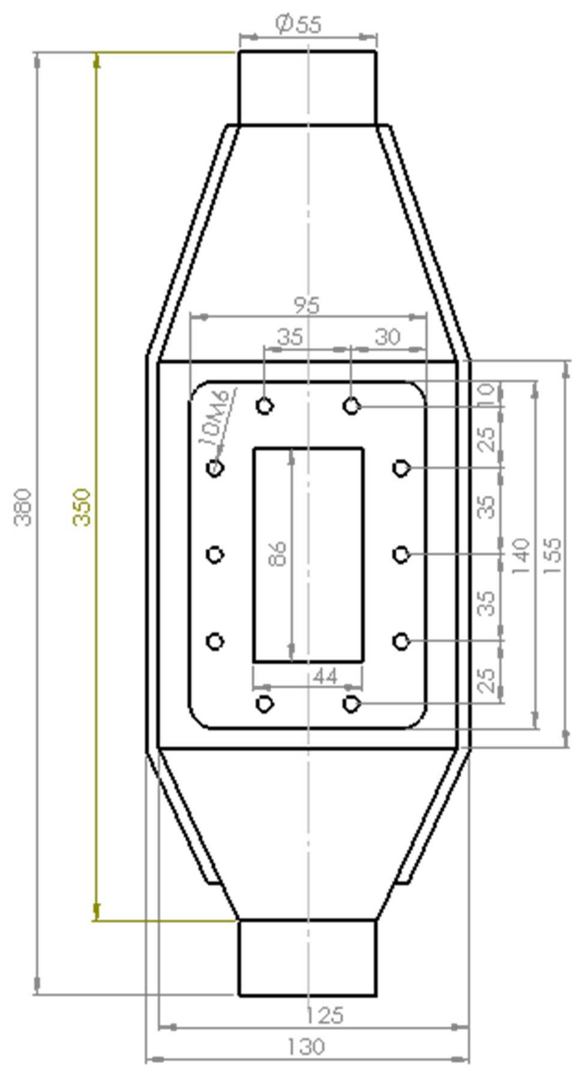

Fig. 4. Overall dimension of the heating chamber and extension of waveguide

The design of new MW-SCR device has been similarly with a commercial after-treatment device in order to better fit to further researches in terms of measurement of the NOx emissions.

The results obtained using microwave heating of ceramic monolith substrate at real scale of SCR device are presented in table below.

The values represents extras of the temperatures that were considered important for the process of selective catalytic reduction initiation (T_{SCR} = 250^0 C) and stability (T_{SCR} = 350^0 C). The graphic representation of temperature evolution is presented, also, in the figure below.

Table 4

Heating time for real scale SCR device

P [W]	t [min]	T [^{0}C]	Ratio [d/h]
1200	1,20	100,00	0,86
	1,31	150,00	
	1,41	200,00	
	1,44	250,00	
	2,14	300,00	
	2,98	350,00	

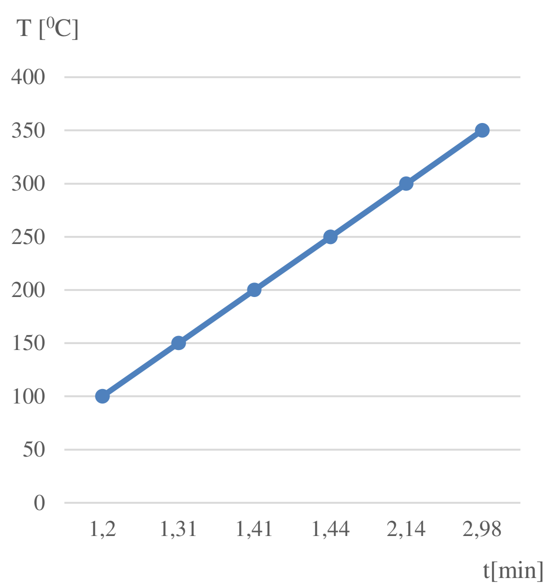

Fig. 5. Temperature evolution in cordierite ceramic substrate for real scale SCR device

According to figure above, the temperature presents a linear evolution. The mathematic model that can be developed using linear approximation is presented in the following equation.

$$T_{SCR} = 50 \cdot t_{SCR} + 50 \quad (6)$$

By comparing the influence of various heating devices that have been developed to improve after-treatment systems, the microwave technology presents the lowest consumption in terms of electrical energy. According to scientific literature, the heating devices using electrical resistance [6, 20] requires around 10,61 minutes for 250^0 C. The microwave technology needs only 1,44 minute which represents a energy efficiency higher than 86%. Similarly for optimum functioning temperature of the SCR device meaning 350^0 C, the heater with electrical resistance needs around 14,85 minutes considering cold start engine. By comparison, the microwave technology needs only 2,98 minutes meaning an energy efficiency higher than 79%.

4. CONCLUSION

Cordierite composite material can be heated in microwave field in order to be used as ceramic substrate in catalytic applications. One of the advantages of using microwave as thermal source in SCR applications is the short heating time meaning around 2,98 minutes. The total heating time is lower than heating time using resistance heating [6].

According to the reported values for resistance heating, the microwave activation of selective catalytic reduction increases the energy efficiency between 79% and 86%. In terms of disadvantages, the microwave heater device has a higher cost than electrical resistance heater and requires additional automation in order to control the heating process. In addition, the microwave heating process of the cordierite is usually unstable and the temperature development in the core of material is patchy in most of the cases. The cordierite material suffers thermal runaway during microwave heating when the injected power of the microwaves is higher than 1200 W.

Cylindrical shape of cordierite is most suitable for microwave heating but the results obtained for micro-scale overall dimensions differ that the results for real scale SCR device even for the similar ratio diameter/height. In case of micro samples the best results, in terms of heating time and stability of the process, were obtained for P1 (ratio 0,49), P2 (ratio 0,34), P4 (ratio 0,78) and P6 (ratio 0,25). However, the total heating time for real scale SCR device (ratio 0,86) reported a lower heating time for 350^0 C compared with P4 (ratio 0,78).

The evolution of the temperature and the occurrence of the microwave plasma discharge during the heating process of the cordierite has required the re-designing of the heating system, by introducing a cooling flange between coupling system and waveguide in order to reduce the local overheating of matching load auto-tuner.

In addition, for real scale SCR device, the re-designing process has included a dielectric ring to avoid contact between cordierite samples and metallic walls of the heating chamber. Figure below presents the MW-SCR after treatment device after re-designing process.

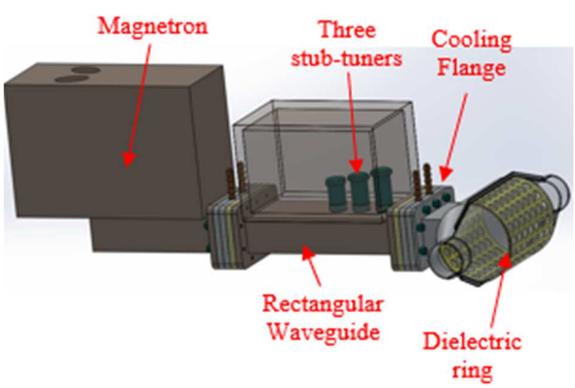

Fig. 5. MW-SCR after treatment system

The dielectric insulation of heating chamber, in order to avoid micro discharges between heating chamber and microwave waveguide, can be done using modern additive manufacturing technologies such 3D Printing (3DP). 3D Printing parameters must be established taking into account that the density/resolution of the printing should be higher.

5. ACKNOWLEDGEMENT

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Cercetări asupra condițiilor de încălzire cu microunde a cordieritei în formă cilindrică pentru aplicații de post tartare

Rezumat: Lucrarea de față propune relizarea unor cercetări asupra condițiilor de încălzire a monolitului ceramic cordierită în câmp unidirecțional de microunde pentru optimizarea proceselor de reducere prin catalizare selectivă a emisiilor de NOx din sistemele de transport nerutiere. Cercetări anterioare au relevat posibilitatea atingerii rapide a temperaturii necesare inițierii reacției de catalizare pentru puteri injectate ale microundelor de 1200 W, monolitele din cordierită având diferite dimensiuni și forme. De asemenea, cercetările au demonstrat faptul că suportul ceramic al catalizatorului interacționează cel mai bine cu microundele atunci când forma acestuia este cilindrică. Lucrarea are drept scop optimizarea mecanismelor de încălzire cu microunde prin expunerea probelor cilindrice având rapoarte diametru/înălțimi diferite.

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