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Technology for manufacturing catalytic systems using a pilot line for precious metal recovery

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The subject of this article is to compare the effectiveness of commercial exhaust gas aftertreatment systems such as TWC and GPF with their prototype variant, which is produced on a pilot line that allows the recovery of precious metals. What's more, the said production line allows the manufacture of components compliant with Euro IV, V and VI standards. Depending on the model of the monolith made, it is possible to reduce the consumption of precious metal raw materials by up to 20%, which should be considered a significant result. The article describes in detail the manufacturing process of metal carriers using the mentioned technology. A dynamic engine dynamometer was used for tests verifying the effectiveness of particulate filters, on which the RDE test route covering the area of the Poznan agglomeration was mapped. The tests performed are particularly important, as it should be borne in mind that according to the forecast in 2025, internal combustion engines powered by conventional fuels will account for 85% of all propulsion sources. In addition, the increasing environmental awareness of vehicle users and manufacturers requires solutions to reduce PM emissions into the atmosphere in both mass and number.

Key words: *DPF, TWC, PM, catalytic systems*

1. Introduction

One of the environmental problems, especially visible in large metropolitan areas, is smog. Its source is, among other things, emissions of harmful compounds from vehicles. Emitted hydrocarbons, nitrogen oxides or particulates, which undergo photo-technical reactions under the influence of sunlight, create photo-technical smog, commonly known as California smog. Reducing emissions from combustion engine units is an essential way to reduce the negative impact of road transport on the environment. Among the components of exhaust gases, particulate matter is the most dangerous to humans. Particularly small particles, which are not filtered by the human respiratory system, are the etiological factors causing disease. The problem of particulate emissions affects both diesel and gasoline-powered internal combustion vehicles. The use of direct gasoline injection contributes to the formation of PM with small diameters (less than 100 nm) [8, 11]. Ultra-fine particles (UFP), despite their small mass and diameter, dominate in number in the ambient air. Significant effects of air pollution by particles with diameters smaller than 100 nm on the occurrence of cardiovascular and respiratory diseases (especially asthma and lung cancer) have been proven [1, 2, 12, 17, 24]. The issue of emissions from dual-fuel vehicles additionally fueled with alternative fuels such as CNG as presented in [27], is discussed. The issue of reducing emissions as a result of replacing conventionally fueled vehicles with alternative vehicles was, in turn, addressed in [25]. Analyzing the literature, it can be noted that researchers focus on analyzing emissions from conventional and alternative vehicles as well as engines for other uses. This may be due, perhaps, to the fact that legislators around the world are systematically reducing emission limits from internal combustion engines [26]. Nowadays, in order to reduce emissions of individual components of exhaust gases, advanced exhaust gas cleaning systems are used [15, 16].

To reduce PM emissions, particulate filters are mainly used. Built on a ceramic or metal support, the filter resembles a honeycomb structure [10]. The inside of the channels are covered with intermediate and catalytic layers. The active layer is made up of Precious Group Metals (PGMs) [20]. These are mainly platinum Pt, palladium Pd and/or rhodium Rh [14]. Oxide catalysts have also found application. The aforementioned active and intermediate layers are applied to the support. Currently, there are two main types of supports – ceramic and metal. The advantages of the first type of carrier are low mass, repeatability of channel shape and limited heat loss. However, the ceramic solution is characterized by high thermal inertia and lower resistance to mechanical damage. Metal carriers, on the other hand, are characterized by high resistance to high temperatures, ease of fabrication of large-diameter components, resistance to sudden temperature changes and, above all, short time to reach light-off temperature. Its disadvantages include high manufacturing cost, significant heat loss during part-load engine operation, the possibility of corrosion, and reduced mechanical strength due to soldering and welding [7]. It is worth noting that the automotive industry is characterized by a very high demand for precious metals. In the case of palladium, the largest amount of this raw material is consumed in the production of catalytic systems used in motor vehicles [22]. As indicated in [22], in 2011, the demand for palladium used in the production of catalytic reactors amounted to 66% of the total demand for this element. One catalytic reactor used in passenger vehicles contains between 3–7 grams of platinum [18]. Over the past five years, a fivefold increase in the price of palladium has been observed. Price increases have also been observed for rhodium and platinum [19]. This directly affects the price of a catalytic reactor, which is a component of the manufacturing price of a new vehicle. In order to optimize the cost and manufacturing process of catalytic systems, an innovative production line was created to recover precious metals. The purpose of this is to reduce the manufacturing costs of these components. This article foretells the results of performance verification tests of prototypes created on a pilot production line.

2. Technology for manufacturing metal carriers

2.1. Manufacturing of metal carriers

Metal carriers are formed by rolling flat and corrugated/perforated metal foil. The thin walls provide a small increase in backpressure at the gas inlet to the carrier and a large OFA surface area. The use of metal foil facilitates the creation of these large-diameter components, which have applications in exhaust gas purification of stationary operating engines (e.g., power generators) and NRMM (Non-Road Mobile Machinery). The manufacture of metal-backed filters is characterized by shorter production times and reduced processes compared to ceramic carriers. The carriers are produced by rolling 0.05 mm thick film. The material undergoes a degreasing process at the beginning of production to maximize surface adhesion properties. Degreasing is carried out in ultrasonic washers (Fig. 1).

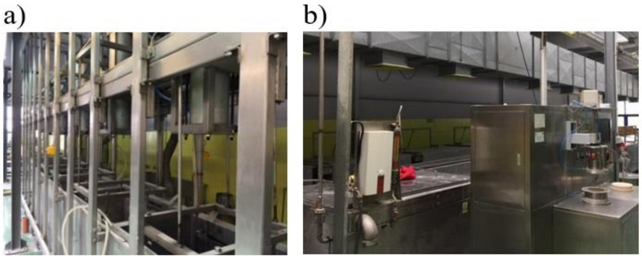

Fig. 1. Treatment of metal film at the beginning of production: a) degreasing in ultrasonic washers, b) coating with catalytic layers together with intermediate layer by bath method

The next stage in the production of the carrier is the spinning process (Fig. 2). Using a feeder with a guide, the metal film is directed to a machine that forms the target shape of the carrier. The film of the required width is then wound onto a spool of a defined diameter and secured with tape before returning to its previous shape (Fig. 3a). The prepared film is coated with catalytic layers together with an intermediate layer by the bath method, less often by spraying. For particulate filters, a coating containing oxidizing elements, mainly platinum, is used. The catalytic layer should be applied uniformly over the entire surface of the carrier. In the filters being developed, the exact composition of the catalytic layer is part of the manufacturer's patent, which determines the proportion of oxidizing or oxidation-reducing elements applied. The catalytic coating varies depending on the type of element produced.

In the case of motor vehicles, the most common shape of the carrier is a cylinder (Fig. 3b). The next stage of production is the manufacture of the housing. The outer part of the carrier and the housing are joined by welding or soldering. This process is carried out manually due to the large differences in the filters and reactors produced. The final stage is the addition of a diffuser at the inlet and a confuser at the outlet of the carrier, depending on the requirements of the customer and the application for a specific use (Fig. 3c).

Fig. 2. Spinning process of metal foil

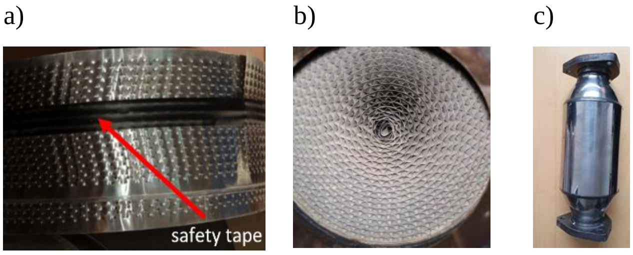

Fig. 3. View of selected stages of metal carrier manufacturing: a) securing the film with tape before restoring the original shape b) cross-section of the finished carrier, c) view of the complete particulate filter

2.2. Recovery of catalytic materials from metal carriers

In the world literature, examples of the application of recycling methods for catalytic reactor carriers and particulate filters made of ceramic materials constitute the vast majority compared to metal carriers. The End-of-Life Vehicles Directive 2000/53/EC sets the general framework for the operation of a country's recycling system, which leaves freedom in the choice of methods and tools used to process vehicles and their components [3, 4]. Pyrometallurgical and hydrometallurgical methods are widely used in the processing of ceramic monoliths. In the case of metal carriers, the recovery of platinides can be carried out using a device equipped with a magnetohydrodynamic pump. Analogous to ceramic monoliths, metal carriers are crushed using dedicated shredders and grinders until a dust with the appropriate grain diameter is obtained. The illustration shows a corrugated film metal carrier for X-ray analysis and recycling (Fig. 4).

Fig. 4. Catalytic reactor metal support used for X-ray analysis and recycling [6]

An analysis of the application of this technology by various research centers indicates that the grinding step of the carrier is not always carried out. The monolith is placed in a liquid metal moving at a certain speed. Two types of pumps are used in the devices. The combination of a magnetic field and current flow creates a force that directs the liquid metal along the axis of the channel (in the case of a conduction pump). In an induction pump, eddy currents are created, which induce a pressure difference at the inlet and outlet of the filter, so that a suction-pressure force is created [5].

According to research conducted by the laboratory of the Institute of Metal Technology of the Silesian University of Technology, the effectiveness of the method was demonstrated by proving the depletion of platinum content in the processed carriers. In Poland, there are more than a dozen plants for the purchase and disposal of all types of carriers. In order to ensure the cost-effectiveness of the process, many companies declare a minimum number of carriers/weights of elements to be delivered for disposal [21]. While processes for ceramic carriers are already fully industrialized and standardized, for their metal counterparts, work is underway to improve the efficiency and effectiveness of the methods used. The undeniable advantage of recycling metal carriers is the degree of material reuse. Fragments of both housing and carrier that no longer contain precious metals are separated and recycled [11]. In recent years, environmental contamination with platinum group metals has been discovered. One likely source is catalytic reactors and particulate filters used in automotive applications. Ceramic supports are subjected to high temperatures, changes in oxidation-reduction conditions and mechanical abrasion. This results in violation of the monolithic structure and leakage of carrier fragments along with exhaust gases into the environment [23]. It is possible to partially eliminate the ingress of PGM metals into the environment by using metallic catalytic supports, which have greater mechanical strength and resistance to rapid temperature changes in the outlet system.

2.3. Innovative technology for manufacturing metal carriers with 20% recovery of precious metals

AWG Polonez has developed an innovative line for manufacturing catalytic systems that meet Euro V and VI standards, which allows the use of 20% recycled precious metals (Fig. 5). The construction of commercial catalytic monoliths consists of spirally coiled or otherwise two types of strips: smooth and corrugated, 0.05 mm thick, bonded together using a binder – hard solder. Such a structure, which resembles a honeycomb in structure, results in a certain number of channels (on average 400 cpsi – the number of holes per 1 cal^2). The exhaust gases flowing through them come into contact with a catalytic layer applied to their surface, which ensures the conversion of CO, HC or NO_x. In the case of AWG Polonez, a catalytic cartridge with a hole density per 1 cal^2 of up to 700 cpsi (600 on average) has been achieved, and the use of hard solder developed at laboratory scale has enabled the use of 0.04 mm thick strips. Washcoat is a porous developed surface containing mainly Al_2O_3, and precious metals derived from the active layer of the catalytic monolith. The active layer contains catalytic substances: palladium, platinum, rhodium or ruthenium. In the technological process of the innovative line, washcoat waste is ground in mills until the desired grain size is achieved, then grain size control is performed. The next step is to produce a homogeneous slurry containing washcoat, precious metals and washcoat from the recovery process. With the use of braze providing sufficiently strong joints, it was possible to use a thinner coil strip to fill the monolith. Braze is a nickel-based eutectic alloy with the addition of phosphorus and boron and chromium. The solder also offers economic advantages due to its reduced melting point compared to current alloys. The fabrication of the catalytic system is divided into 3 stages: preparation of the raw materials needed to make the catalytic monolith and ultimately the finished product-catalyst, physical and chemical processes of the monolith taking into account recycled materials, incorporation of the monolith into the finished product. A key element of monolith manufacturing is the brazing of monolith components. Due to the requirements of the process, it is carried out under a 10-3Tr vacuum. A nickel-based alloy with the addition of phosphorus and boron will be used as the binder for brazing. The alloy has a liquidus temperature below 900^{o}C. This will make it possible to reduce the temperature of the process, which is beneficial for economic as well as environmental reasons. A laboratory tube furnace was used to carry out tests on joining stainless steel layers with brazing. Observations of the structure of the brazed joint were carried out on an X-ray microanalyzer. The obtained joints were characterized by about 90% gap filling by the solder material, while the American Welding Society for responsible joints considers 85% coverage of the bonded surfaces sufficient. The evaluation of the quality of the solder joints, made on the basis of macroscopic observations of the shapes of the solder outflows, also made it possible to describe them as correct. In most of the connections, correct concave-shaped efflorescences were observed, proving good adhesion of the solder to the parent material. In all samples, regardless of the soldering parameters used, the joint structures were qualitatively similar. The native material and the zone of the actual joint could be distinguished in the tested joints. Strength tests confirmed that the tested binder in the form of an amorphous strip meets the requirements for solder joints in catalytic monoliths. The very good performance of the used braze allowed for saving 20% of material in the actual process.

Fig. 5. Innovative metal carrier production line with 20% precious metal recovery

3. GPF filter testing

3.1. Description of the manufactured filter

The metal-backed particulate filter made on the innovative production line was a flow-through filter. It consisted of 50 filter channels with a height of 2 mm, which translated into 200 cpsi. One of the basic geometrical parameters of the filter media is the angle of perforation of the metal film. The prototype filter was characterized by dividing the length of the carrier channel into 3 sections (Fig. 6). Each with a length of 50 mm, but variable perforation angle: 20°, 25°, 30°. The use of perforations of the carrier's metol film forced the flow of exhaust gases on the outer radii of the carrier's longitudinal axis, which increased the efficiency of the filter (Fig. 7a). The diameter of the carrier was 100 mm, the length was 150 mm (Fig. 7a and 7b). These dimensions are close to the solutions used in series for the tested engines by manufacturers of catalytic reactors and particulate filters in the vast majority of solutions. The angle of inclination of the diffuser and confuser (30°) was adjusted to the diameter of the exhaust system pipe.

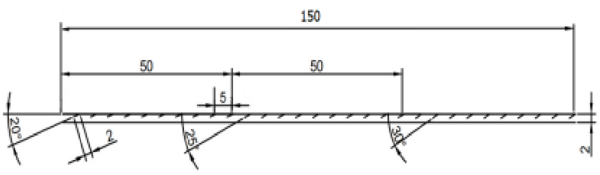

Fig. 6. Geometry of the channel of the prototype filter

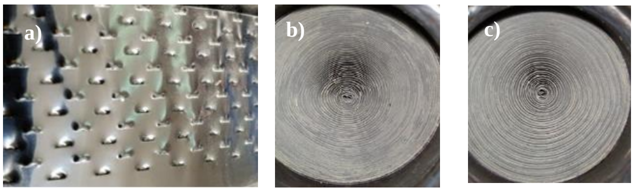

Fig. 7. Prototype filter: a) perforations of metal sheet, b) filter inlet, c) filter outlet

3.2. Methodology

Verification of the efficiency of the manufactured filter was carried out by measuring the emission of harmful exhaust gas compounds downstream of the exhaust gas aftertreatment system. The subjects of the study were 4 components of the exhaust gas cleaning system. Two commercial solutions were used in the study – a TWC catalytic reactor and a GPF/TWC particle filter, as well as a prototype catalytic coated particle filter manufactured on the innovative production line described earlier. The prototype solution was juxtaposed with the TWC 400 CPSI reactor (Fig. 8). Data on the individual research configurations are summarized in Table 1.

Table 1. Configurations of exhaust gas purification systems used for testing

No	Number of elements	Distribution of elements	Number cpsi	Type configurations	Material carrier
1	1	TWC	200	commercial	ceramics
2	1	GPF/TWC	400		
3	2	TWC + GPF/TWC	400 + 200	prototype	metal
GPF/TWC – catalytic coated particulate filter					

The test was carried out using an engine dynamometer. The tested configurations of exhaust gas treatment systems were mounted behind an engine meeting the Euro 5 emission standard. The location of the various components on the bench is shown in Fig. 9.

A dynamic braking stand was used to load the engine. The measurement was carried out on the basis of the author's test, which reflected the test in real operating conditions. The premise of the test was to simplify and shorten the test procedure performed under real conditions to tests using an engine dynamometer. The test was divided into 3 cycles corresponding to the division considered in the RDE procedure. The dynamometer used is characterized by a short overdrive (load change) time, making it possible to reproduce a previously recorded test, as well as to reproduce the braking of an engine that is driven by an electric unit. The simulated speed of the vehicle is shown in Fig. 10. According to the standard, the parameters in each of the three cycles are strictly defined by the time the vehicle travels at a speed equal to or less than 60 km/h for the urban cycle, between 60 and 90 km/h for the extra-urban cycle, and between 90 and 140 km/h for the highway cycle. A mobile apparatus from the Portable Emission Measurement Systems (PEMS) family was used to measure exhaust emissions of harmful compounds. Emission rates of gas compounds (CO, CO_2, NO_x, HC and O_2) and exhaust mass flow rates were measured using the SEMTECH DS apparatus. PM_m concentration was measured using AVL's MSS (Micro Soot Sensor) analyzer. The method of operation and parameters of the measurement apparatus used are described in detail in other publications by the authors [9, 13, 28, 29].

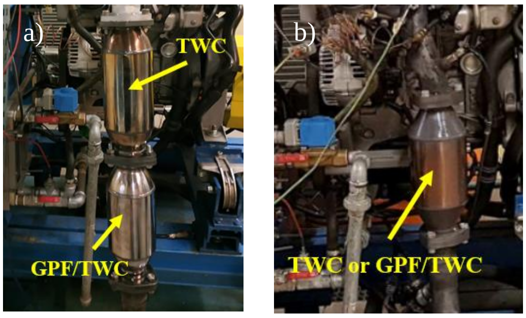

Fig. 8. Arrangement of the outlet system components depending on the tested configuration: a) TWC+ prototype GPF/TWC reactor, b) commercial TWC or GPF/TWC

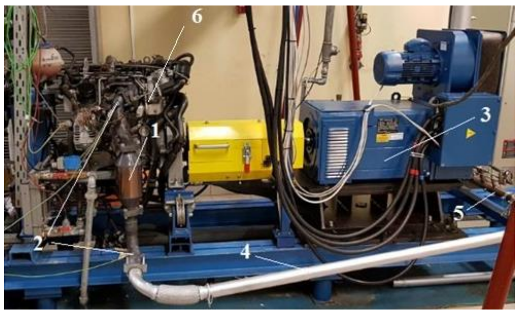

Fig. 9. Test stand: 1 – particulate filter, 2 – temperature measuring points, 3 – electric motor of dynamic dynamometer, 4 – exhaust system, 5 – exhaust gas flow meter, 6 – internal combustion engine

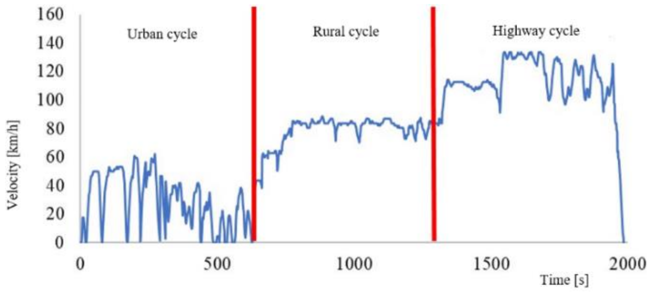

Fig. 10. Vehicle speed in the author's engine dynamometer test

4. Research results

4.1. Configuration 1

The choice of test methodology is based on the need to develop a solution that combines the advantages of on-road and laboratory testing. The first exhaust aftertreatment system configuration tested consisted solely of a commercial TWC (Fig. 11).

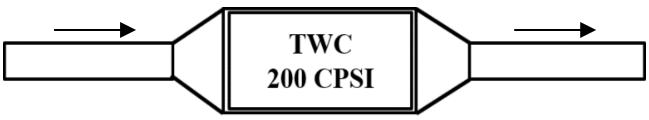

Fig. 11. Schematic of test system with commercial ceramic carrier

Characteristics of PM_m emission intensity over a range of load and crankshaft speed illustrate ranges with increased emission intensity: a load of 5% for speeds in the 1000–1250 rpm range, loads of 10 and 20% for speeds in the 3000–3250 rpm range, and a load of 30% for speeds in the 2250–2750 rpm range. In addition to the large value observed for the engine's idling points, particle emissions under conditions corresponding to the entire load range and higher speeds account for the largest share of the entire test (Fig. 12). Most PN were emitted during engine operation at medium and high speeds in the partial load range (Fig. 13).

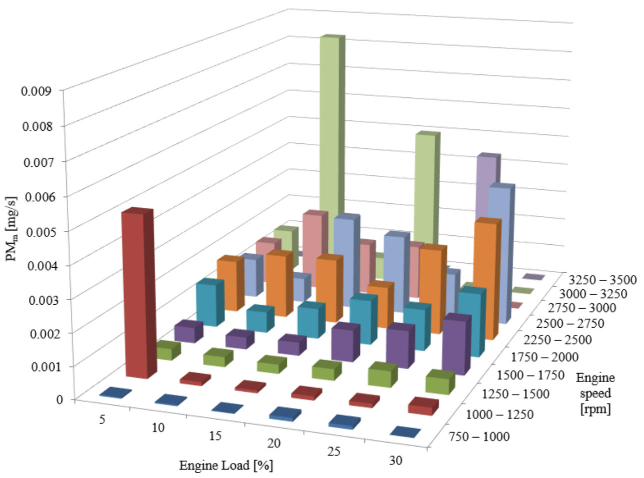

Fig. 12. PM_m emission intensity in the range of engine speed and engine load for configuration 1 of the exhaust system in the rendered test

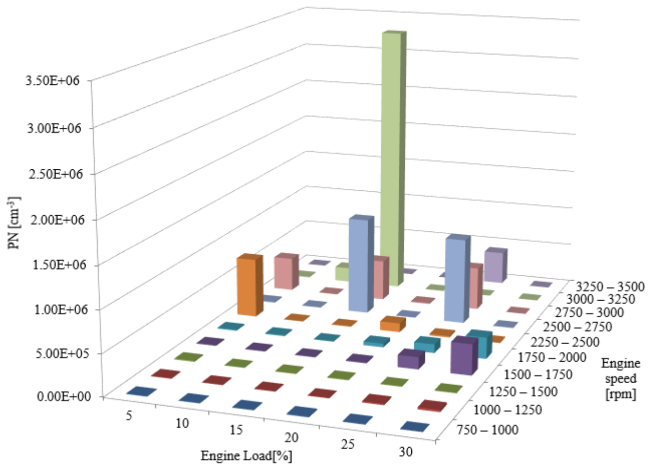

Fig. 13. PN concentration in the range of engine speed and engine load for configuration 1 of the exhaust system in the rendered test

4.2. Configuration 2

Another configuration tested was a commercial GPF/TWC filter in series on the engine accepted for research (Fig. 14).

Fig. 14. Schematic of the test system with commercial *wall-through* ceramic carrier

The intensity of PM_m emissions over a range of load and crankshaft speed is divided into three ranges. The first reflects engine operation with a load of 5–30% and an engine speed of 750–1250 rpm (Fig. 15). The highest value in this range was registered for the lowest value of engine load and speed corresponding to idling. The other values are several times lower, indicating the effect of increasing the load on increasing the efficiency of the particulate filter. In the second interval of 1250–2750 rpm, emissions are uniform. There is a several-fold increase in the recorded values in the third interval, 2750–3250 rpm.

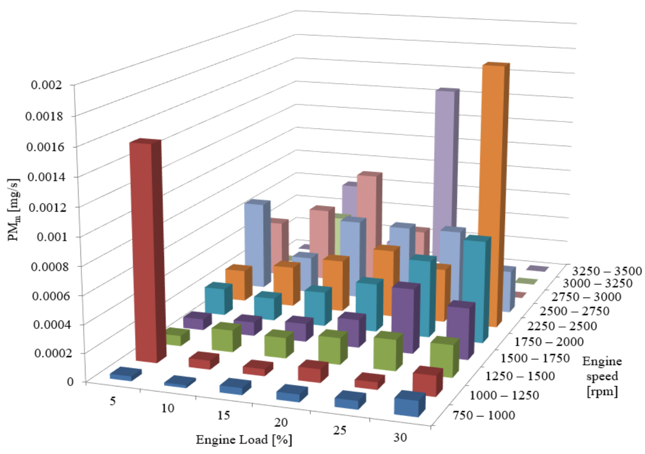

Fig. 15. PM_m emission intensity in the range of engine speed and engine load for configuration 2 of the exhaust system in the rendered test

PN concentration is highest in the speed range above 2000 rpm and load above 20% (Fig. 16). From the characteristics, it can be seen that low rotational speeds have an aggregately smaller effect on PN emissions compared to near-maximum speed values.

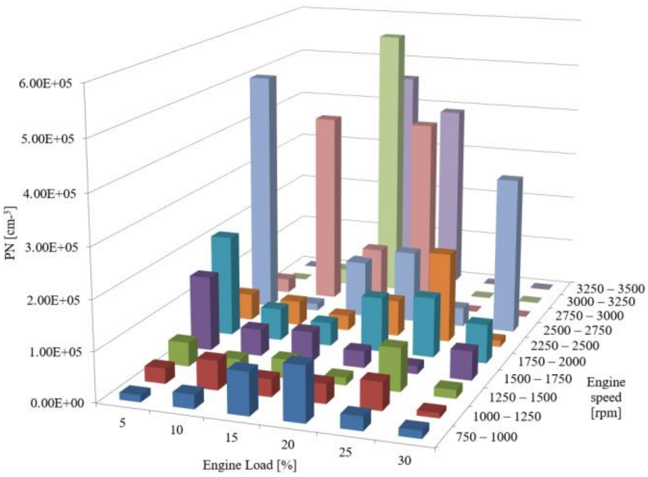

Fig. 16. PN concentration in the range of engine speed and engine load for configuration 2 of the exhaust system in the rendered test

4.3. Configuration 3

The premise of Configuration 3 was to place two elements made with metal carriers in the exhaust system. The first was a TWC 400 CPSI reactor supporting CO, HC oxidation and NO_x reduction reactions. The second element was a prototype GPF/TWC acting as a filter (Fig. 17).

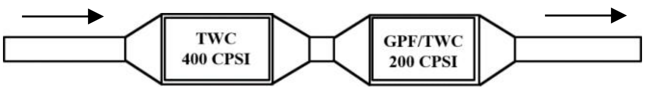

Fig. 17. Schematic of exhaust gas purification system with TWC 400 CPSI reactor and prototype GPF/TWC 200 CPSI metal carrier

The maximum value of PM_m emission intensity differs from the other results by about ten times. This result was obtained for a load of 5% and a rotational speed in the range of 1250–1500 rpm (Fig. 18).

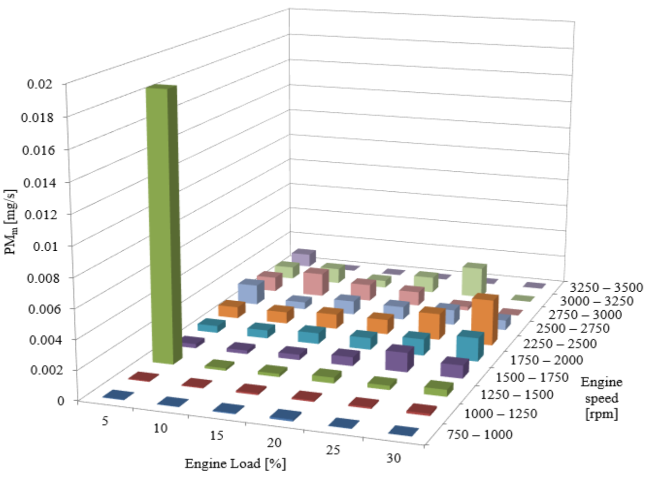

Fig. 18. PM_m emission intensity in the range of engine speed and engine load for configuration 3 of the exhaust system in the rendered test

These values correspond to operation in urban conditions during congestion. The intensity of PM_m emissions in such a case is particularly undesirable due to the exposure of people near the roadway. PM_m emission intensities above 1500 rpm reach similar values for the entire load range. The highest PN was measured during low-load operation in the crankshaft speed range from 1500–1700 rpm (Fig. 19). Apart from this case, the effect of simultaneously increasing speed and load resulted in a gradual increase in PN until it reached values corresponding to engine operation at the highest vehicle speeds.

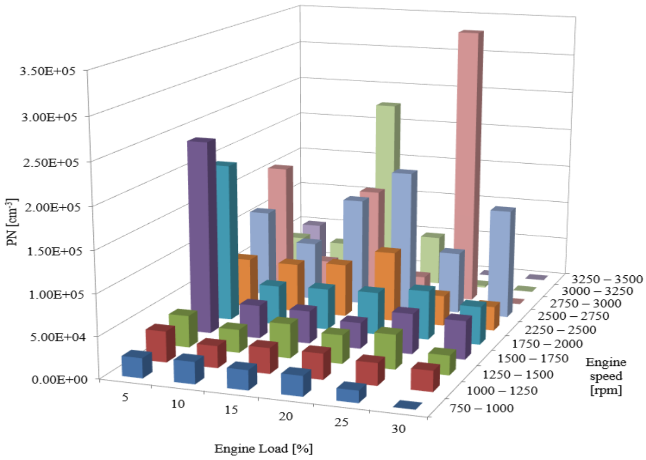

Fig. 19. PN concentration in the range of engine speed and engine load for configuration 3 of the exhaust system in the rendered test

In order to summarize the results obtained, graphs were made showing the measured values for the various configurations that were tested (Fig. 20).

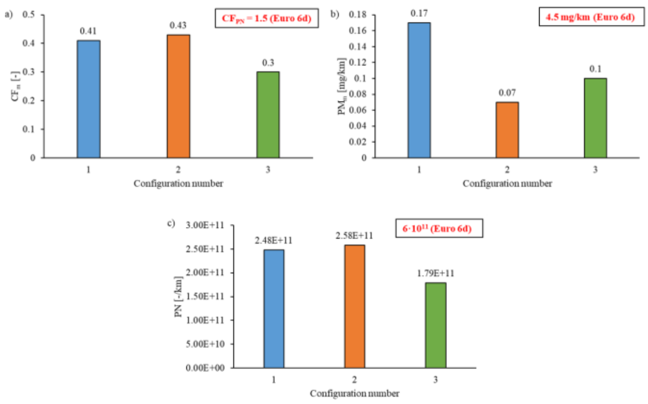

Fig. 20. Summary of the results obtained for each configuration: a) CF_{PN}, b) PM_m, c) PN

Based on the Conformity Factor for PN, it can be concluded that all test facilities did not exceed the limits assumed for the Euro 6d standard. The particulate filter manufactured on a state-of-the-art assembly line allowing recovery of precious metals had the lowest value, which means that among the tested configurations it was the most effective. Analyzing the measured values of particulate mass analogously to the first chart also, none of the configurations exceeded the limits. Both the second and third variants reached values close to each other. The highest value was recorded for configuration number 1, in which a commercial trifunctional catalytic reactor was installed. In the case of the third graph showing the number of particles emitted, the prototype particulate filter was also the most effective. Analyzing the other commercial solutions, there is an increase in particle number emissions of about 30% than for configuration number 3.

5. Conclusion

Three configurations of the exhaust gas treatment system were tested, and their effectiveness was compared in a test that replicated a real-world run complying with the requirements of the RDE procedure. All configurations met the requirements of the Euro 6d standard for PM_m and PN emissions. In configuration 3 used, the measured CF and PN values are about 30% lower than those of commercial exhaust aftertreatment systems. This is directly related to the construction of the described filter and the solutions used in it. Therefore, the manufactured prototype particulate filter in a state-of-the-art pilot line enabling precious metal recovery has better particulate filtering properties than commercial solutions. What's more, the innovative manufacturing technology makes it possible to use metal carriers with 20% precious metal recovery, while meeting the respective guidelines for Euro V and Euro VI standards. Tests of commercial carriers were carried out to compare performance in reducing gaseous components of exhaust gases and filtration efficiency with prototype configurations made. Based on the test results, it is clear that the use of metallic media as replacements for commonly used ceramic media filters will be a more effective solution.

Acknowledgements

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Unia Europejska Europejski Fundusz Rozwoju Regionalnego

Nomenclature

CF	conformity factor
CO	carbon monoxide
CO_2	carbon dioxide
GPF	gasoline particulate filter
HC	hydrocarbon
NO_x	nitrogen dioxide
O_2	oxygen
PEMS	portable emission measurement system
PM_m	particulate matter mass
PN	particulate number
RDE	Real Drive Emission
SI	spark ignition
TWC	three way catalysts

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