Development of a Laboratory Test for the Deposit Forming Tendency of Diesel Fuels

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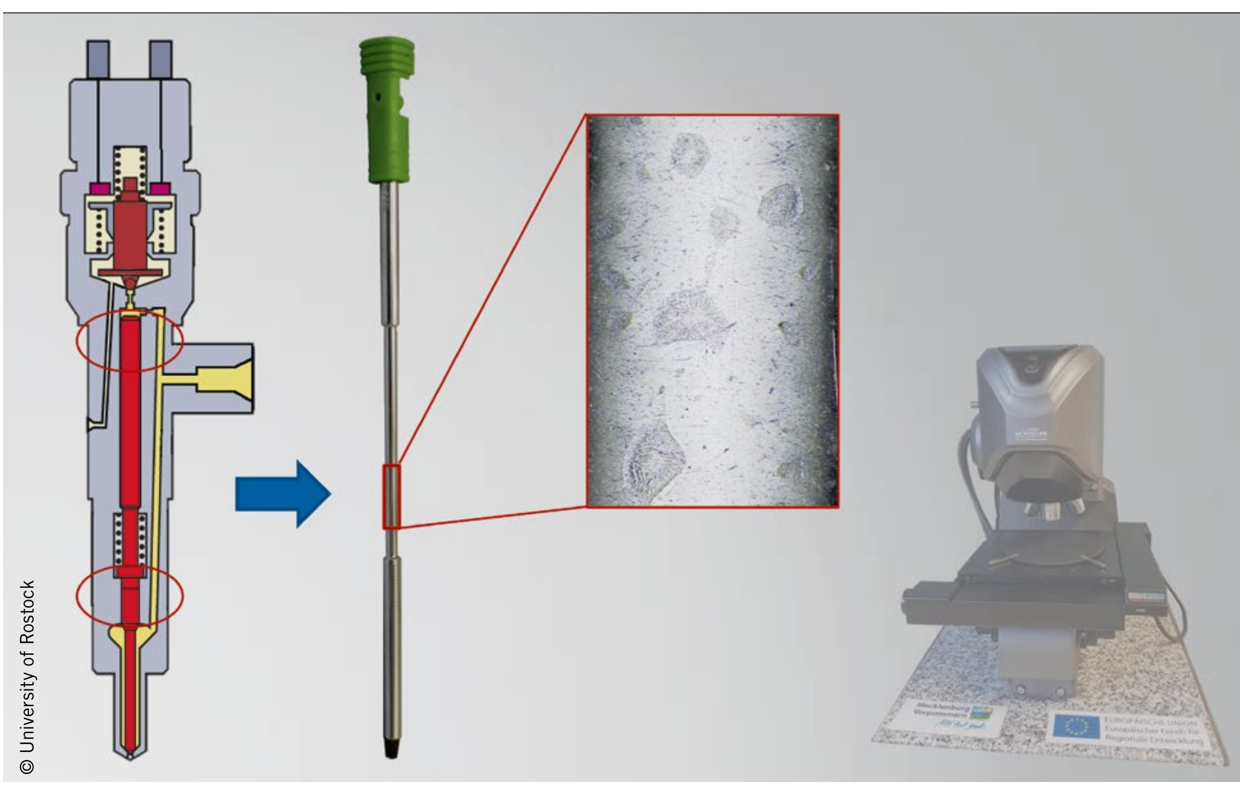
In recent years problems have been reported in engine operating characteristics and in dynamic injector behavior related to fuel-based deposits inside common rail injectors. The reasons for the formation of deposits are very complex and include both fuel-side and design influences. Cause clarification can often no longer be achieved with complex and expensive engine tests/injection bench tests. Therefore, there is a high demand for a simple and widely available laboratory test for preventative fuel screening. At the Department of Piston Machines and Internal Combustion Engines of the University of Rostock, in a FVV research project a test method was developed to evaluate the deposit forming tendency of diesel fuels inside of common rail injectors.



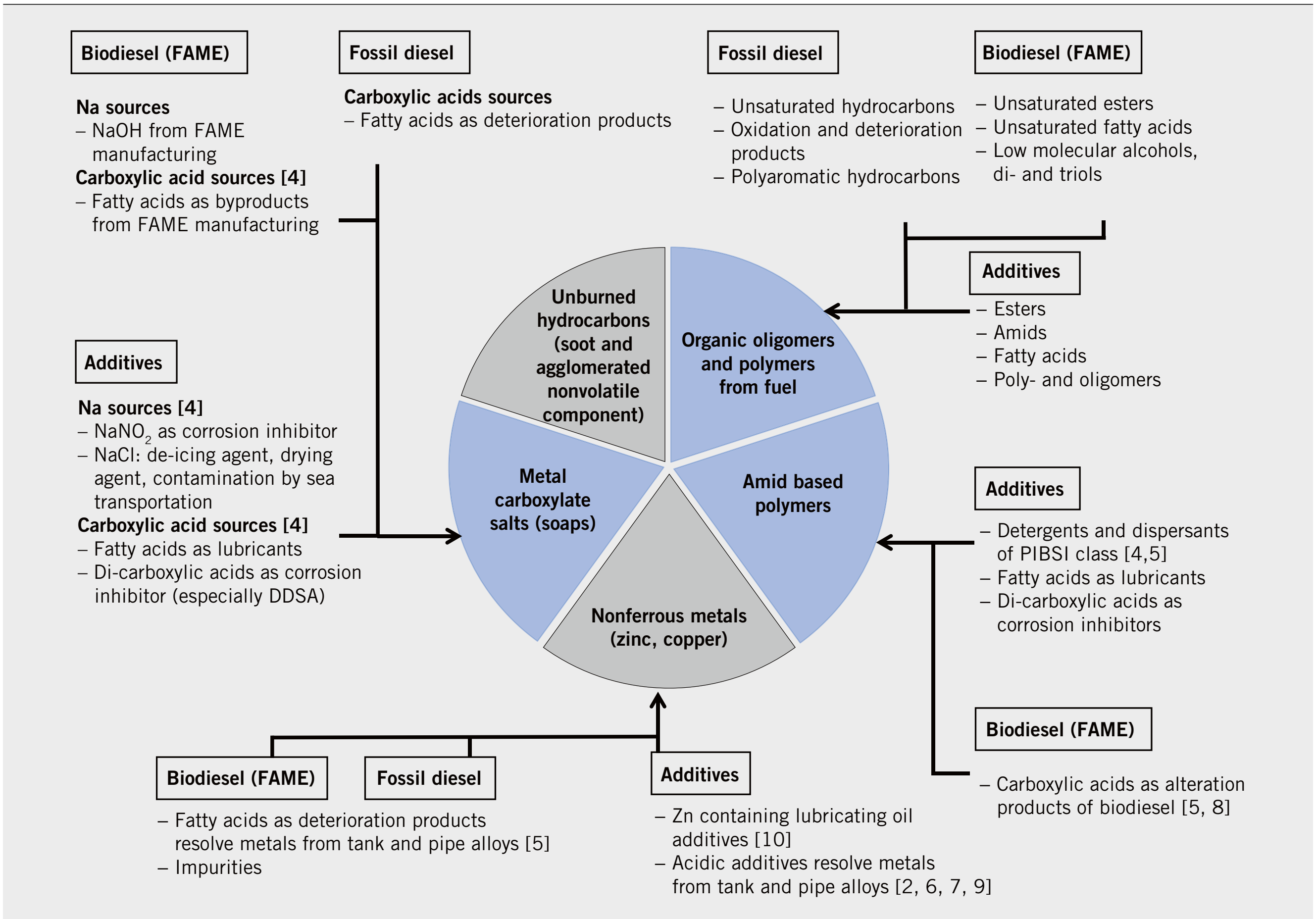


FIGURE 1 Deposit types and sources [1] (© University of Rostock)

1 MOTIVATION

The introduction of stricter emission legislations requires a steady development of the diesel engine combustion process. The trend goes towards increasingly complex components; whose secure functionality is only possible by adhering to extremely narrow tolerance limits – especially in the area of guides of moving parts. At a typical needle clearance of HD-CR injectors of approximately 2 to 4 µm, even deposits of a few micrometers can have a considerable influence on the operating behavior of injectors. Conceivable consequences would be increased emissions and rough engine running due to impairments in the timing of the injector or changed injection quantities as well as severe engine damages due to permanently incorrectly injecting injectors or jamming needles/ valves. Common rail injectors for heavy-duty engines are particularly in the focus of attention because of their special requirements. These engines are characterized by applications with the highest rail pressures, a high proportion of high-load operation and use in a wide variety of regions and markets. In addition, high reliability for a long service life and flexibility in terms of various fuels are required. However, the results gained in the project can also be transferred to passenger car injectors.

2 STATE OF KNOWLEDGE

The literature reports a variety of different deposit sources and reaction pathways for the formation of reactive and deposit-forming fuel components [1–3]. They often appear in combinations that differ due to regional, fuel and additive factors. **FIGURE 1** shows an overview of the observed and investigated deposits as well as the cause of these deposits, whereby the deposition types of the high lighted segments are explained in more detail below. It is apparent how complex the deposit formation can be and that a variety of deposit forming components is causally responsible. The causes extend both to the influence of the base fuel as well as diverse interactions of additives. Therefore, the further development of the diesel-injection systems must be accompanied by a further development of the fuels. Soap and amide-type deposits are considered to be particularly injector-critical, as they already lead to considerable restrictions on the injector function on the test bench after a short time.

FIGURE 2 Demonstration device (© University of Rostock)

3 METHOD DESCRIPTION

For the development of the test a demonstration device from PAC – Walter Herzog was available, **FIGURE 2**. The device was originally developed to evaluate the high-temperature oxidation stability of kerosene (Jet Fuel Thermal Oxidation Test according to ASTM D3241). Therefore, a hardware-side adaptation of the device to the higher viscosities of diesel fuels (up to 5 mm²/s) as well as the sealing materials used (biodiesel compatibility) was required. In addition, principally new evaluation methods had to be developed for the deposits, since especially the particularly critical soap-like deposits are hardly visible to the naked eye.

The evaluation of the test results in the Diesel Deposit Formation Test (DDFT) takes place by means of layer thickness measurements of the deposits on the surface of an Al-heater tube, **FIGURE 3**, by means of an ellipsometer. Furthermore, the blocking speed of a filter with a pore size of 17 µm is monitored by pressure drop. This filter is installed in the experimental setup as shown in **FIGURE 4**. The essential work steps for performing the DDFT test are briefly summarized below: The specially manufactured and commercially available Al-heater tube is installed in the heating tube jacket (see below), prefilter and test filter are mounted and the thermocouple is placed inside the heating element. Potential deposits are formed on a cylindrical measuring surface (3.175 mm diameter x 60 mm length) between the shoulders of the tube. The fuel inlet to the heating element is at the 0 mm position and the fuel outlet at 60 mm, **FIGURE 4**.

The fuel sample (600 ml) is placed in a sample reservoir and purged through with dried air for 6 min. The fuel system is then pressurized to 34 bar and the test tube is heated resistively to the set point temperature of 240 °C selected in the program. The temperature is controlled by a thermocouple, which is positioned inside the heater tube (set point temperature T_{max} at 39 mm). An isocratic pump moves the fuel through the measuring system at a defined flow rate (3 ml/min), **FIGURE 5**. The fuel flows around the heater tube and is directed through the precision filter into the waste container. The test duration is 150 min.

Oxidative deposits are typically observed at the hottest area of the test tube, which is between the 30 mm and 50 mm position. Soapy deposits are mainly located in the low temperature range (up to 180 °C). Deposits (fuel degradation products), which are stripped away by the fuel flow and entrained, lead to the blocking of the test filter. After the end of the test, the measuring arrangement will be disassembled, lines and test tubes rinsed with heptane, dried and later evaluated optically.

4 EVALUATION OF THE MEASUREMENT RESULTS

The following methods were used for the evaluation of the measurement results:
– ellipsometry (layer thickness measurements)
– digital and laser scanning microscopy (overview and detailed images of the deposits)
– FTIR microscopy (identification of the deposits).

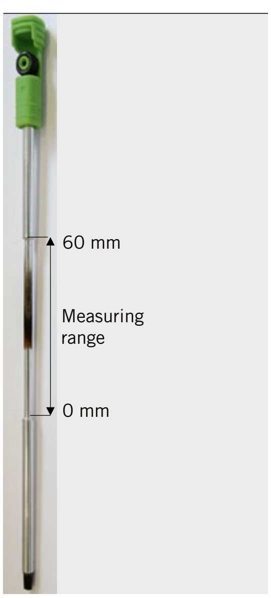


FIGURE 3 Aluminum heater tube (© University of Rostock)

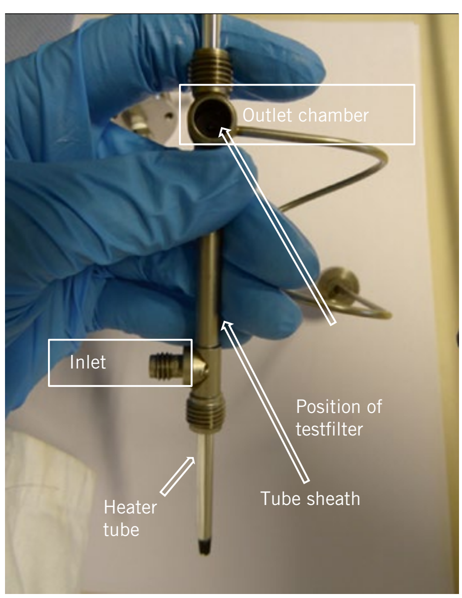


FIGURE 4 Installation of heater tubes (© University of Rostock)

FIGURE 5 Measuring system (© University of Rostock)

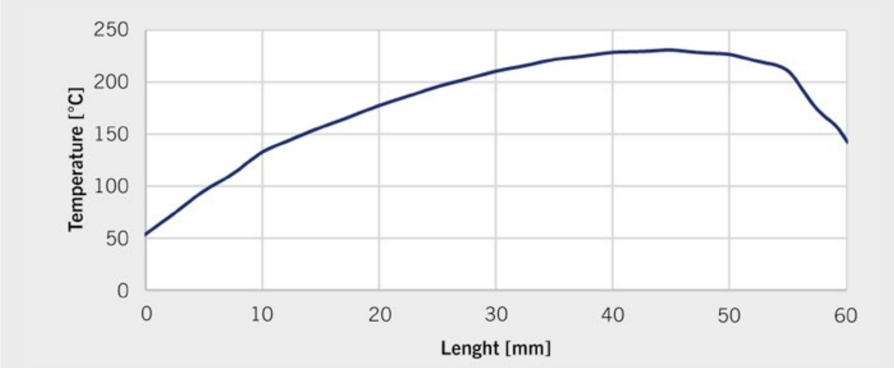


FIGURE 6 Temperature profile on the heating tube surface (T_{max} 240 °C) (© University of Rostock)

Reference diesel fuels for DW10C test:

- RF 79-07 (reference fuel – without any effect in the engine test)
- RF 79-07 + 0,5 ppm Na + 10 ppm DDSA (reference fuel with soap forming substances)

Test number	Na [mg/kg]	Dodecenyl succinic-acid [mg/kg]	Δp [mmHg]	Time to 25 mmHg [min]	
1	0	0	0	0	No restictions in the DW10C test
2	0	0	0	0	
3	0.5	10	0	0	Cold start problems in the DW10C test
4	0.5	10	0	0	
5	3	20	**250.1**	**52.8**	Harder test conditions
6	3	20	**246.6**	**49.8**	

FIGURE 7 Overview of the test fuels and measured differential pressures from DDFT (© University of Rostock)

The combination of data from pressure increase during the DDFT test, evaluation of the layer thickness measurement data (standard spot thickness and thickness maps) by means of ellipsometer in comparison to the recorded digital microscope images of the heater tubes with 100 times magnification, allows a reliable assessment of the generated deposits. In selected cases FTIR microscopic measurements were carried out to confirm the assumed type of deposits.

5 DETERMINATION OF THE TEMPERATURE GRADIENT ON THE HEATER TUBES

Another important criterion for assessing the formation of deposits is the temperature. Since it is known from literature that certain deposit types occur in certain temperature ranges, the determination of the temperature profile on the surface of the heater tube was of significant importance for the interpretation of the results. The determination of the temperature gradients is based on simulations because of the poor accessibility for thermocouples on the heater tubes in the DDFT.

From injection bench tests it is known that typical maximum fuel temperatures inside common rail injectors are between 150 and 180 °C. Therefore, the set point temperature of 240 °C was chosen, that on one hand this temperature range is covered on the surface of the heater tubes and on the other hand, that no excessive thermal-oxidative decomposition of the fuel appears. The boundary conditions of the simulation taking into account the temperature-dependent material properties were adapted to this temperature. Based on the simulation, the following temperature curve results on the heater tube surface, which is the basis for the further testing, **FIGURE 6**.

6 SELECTED RESULTS AND THEIR VALIDATION AT THE INJECTION TEST BENCH

In order to evaluate and compare the deposit formation tendency of different diesel fuels in the DDFT test and injection bench rig, a test fuel with defined composition is tested. This test fuel (CEC RF 79-07) is used as fuel in the IDID DW10C engine test (CEC F-110-16) and shows no abnormalities in this test. That means, the RF 79-07 does not lead to any restrictions in injector operation. In order to produce a targeted formation of soap-like deposits in the DW10C engine test, the reference fuel is admixed with the soap forming substances DDSA (dodecenyl succinic acid) and an organic Na-component. With regard to the measured differential pressure in DDFT, both reference fuels from the DW10C engine test initially remained inconspicuous. Only an increased concentration of soap formers led to a noticeable increase in pressure, which led to the opening of the bypass circuit after approximately 50 minutes (filter blocking due to soap crystallization), **FIGURE 7**.

Looking at the results of the layer thickness measurements, they initially show a surprising result. The standard spot thickness (mean deposit thickness of the thickest 2.5 mm² area as defined in ASTM D3241) indicates higher layer thicknesses for the no-effect reference fuel than for the injector-critical fuel with the addi-

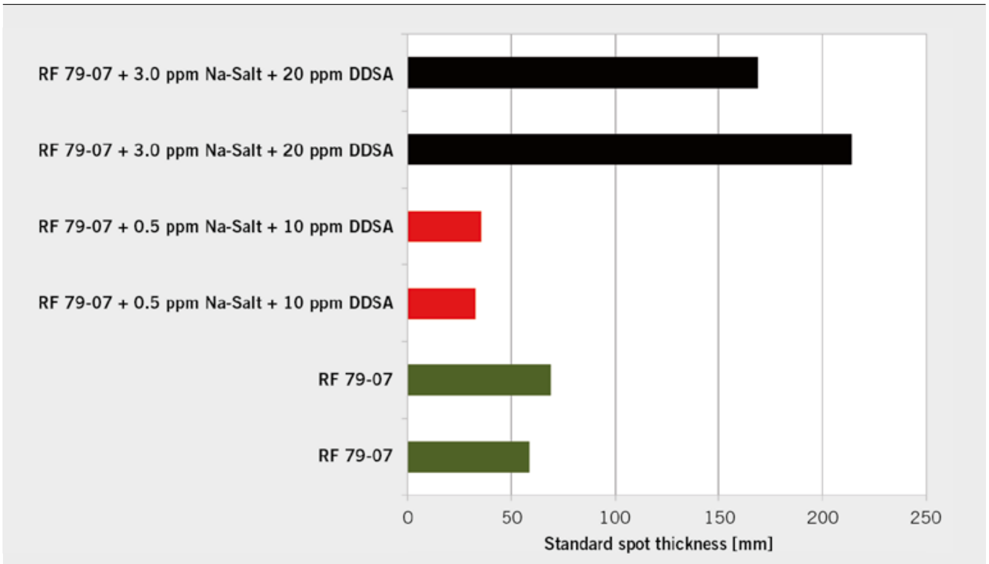


FIGURE 8 Standard spot thickness of the deposits without and with the addition of soap formers to the reference DK 79-07 (© University of Rostock)

tion of soap forming components, **FIGURE 8**. However, looking at the associated ellipsometer thickness maps, it can be seen that the deposits are formed in different areas of the heater tube surface. The deposits of the no-effect reference fuel are formed on the hot part of the heater tube and result from the fuel oxidation at high-temperatures. With soap-forming components the deposits are located in the low-temperature area of the tube. Simultaneously, the formation of deposits in the high-temperature range is prevented by the cleaning effect of the soaps, **FIGURE 9**. A temperature dependent evaluation of the deposits, that means, if injector-related temperature ranges are considered, shows, that the high-temperatures at which the fuel oxidation begins are not reached in the real injector. Deposits in this temperature range are not relevant to the injector.

At a higher dosage of the soap formers individual conglomerates were observed with high layer thickness, but no longer a closed layer. It can be assumed that it comes to a removal/debonding by passing fuel. Investigations on the injection system test bench impressively prove the correctness of this assumption, **FIGURE 10**. The no-effect reference fuel is absolutely unremarkable in the bench test. If soap formers are added to the reference fuel, noticeable deposits with very high layer thicknesses in the µm range are observed on the injector components. During the injection bench test large variations in the leakage temperature (indicator for deposit formation/destruction) and injection quantity variations (injection progress indication), indicate a malfunction of the injector. Both on the heater tube and on the injector intermediate plate, soaps were detected by their typical bands by FTIR microscopy. A similar result can be seen when using another fossil reference fuel (reference DK 0). It could be shown that the developed test is very sensitive regarding soap forming components in fuels (0.5 ppm Na) and that the results correlate very well with the results of injection test bench tests. The new, temperature-dependent assessment of the layer thicknesses in the DDFT is

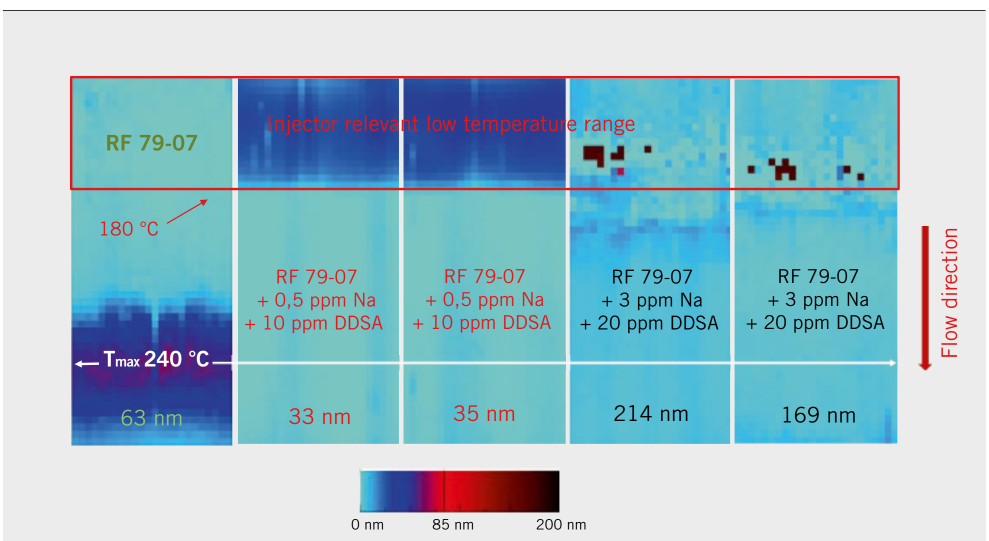


FIGURE 9 Thickness maps of tubes with soap-like deposits (© University of Rostock)

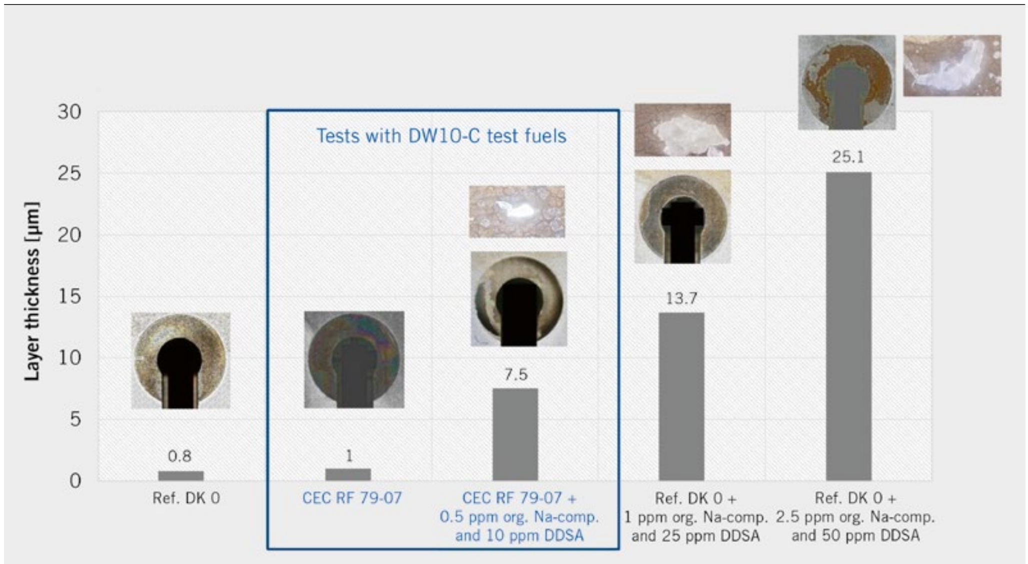


FIGURE 10 Layer thicknesses on injector components (intermediate plates) after tests with DW10C reference fuels at the injection system test bench [11] (© University of Rostock)

intended to enable future classification or meaningful fuel selection with regard to the robustness and application fields of the specific injectors.

7 SUMMARY

On the basis of commercially available test equipment (Jet Fuel Thermo Oxidation Tester and Ellipsometer), a bench test independent method for the assessment of the deposition tendency of diesel fuels (Internal Diesel Injector Deposits) was developed and tested, in particular with regard to the preventive screening of diesel fuels. For this purpose, a JFTOT device was adapted to the diesel fuel analysis and the method was modified so that the test conditions reflect relevant temperature ranges and fuel flow rates of current injector systems. The obtained results correlate very well with results from injection bench tests. The DDFT sensitively indicates different types of fuel related deposits and has the potential to replace expensive test bench tests in the future.

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