Thermal Barrier Coatings for Heavy-Duty Diesel Engines

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Opponent is Professor Uta Klement from Chalmers University of Technology, Sweden
Abstract

Due to increasing concern about climate change and stricter regulations for greenhouse gas emissions, there is great interest in reducing CO₂ emissions from heavy-duty diesel engines. Thermal barrier coatings (TBCs) can be used for insulating engine components, thereby reducing the heat losses from the engine and potentially improving its efficiency. The reduced thermal load on the components is also beneficial for the engine lifetime. So far, the success of TBCs in diesel engines has been limited, due to lifetime issues, lower heat loss reductions than anticipated, and deteriorated combustion. For the purpose of reducing the heat losses from diesel engines, the overall research objective of this thesis is to evaluate TBCs with respect to lifetime and thermal insulation properties under conditions similar to those of a heavy-duty diesel engine.

Thermal cycling fatigue tests show that the type of microstructure greatly affects the lifetime of yttria-stabilized zirconia (YSZ) TBCs when these are exposed to conditions typical of heavy-duty diesel engines. A segmented atmospheric plasma-sprayed (APS) coating displayed the best performance of the APS coatings, only outperformed by a plasma spray–physical vapour deposition coating. When comparing the thermal cycling fatigue life of different top coat materials, it was found that YSZ performed best.

The type of microstructure of plasma-sprayed YSZ coatings greatly influenced the thermal conductivity and hemispherical reflectance, when evaluated at temperatures and thermal radiation wavelengths typical of the combustion chamber of a diesel engine. The coating with the best properties for the combustion chamber was the nanostructured APS coating, followed by the conventional APS coating, due to a combination of low thermal conductivity and high reflectance at temperatures up to 600°C. Hemispherical reflectance was not improved by adding a thin metallic surface layer.

In situ heat flux measurements of plasma-sprayed TBCs inside the combustion chamber showed that gadolinium zirconate (GZ) was the top coat material that provided the best thermal insulation, while YSZ coatings with different types of microstructure and lanthanum zirconate were less effective. YSZ with the pores sealed with aluminium phosphate had higher heat fluxes than those of a steel reference. The heat flux measurements also showed that there is a running-in period for plasma-sprayed YSZ and GZ coatings inside the combustion chamber. The heat flux through these two TBCs decreased during the first 2–3 h before stabilizing.

When deposited on pistons, suspension plasma-sprayed GZ was the TBC that displayed the best results in an engine test, reducing the temperature of the piston cooling oil and increasing the exhaust temperature. However, there was no improvement in the efficiency of the engine.

Keywords
Thermal barrier coatings, diesel engine, thermal cycling fatigue, heat flux, running-in, exhaust manifolds, yttria-stabilized zirconia, gadolinium zirconate, lanthanum zirconate
Sammanfattning

Ökande oro för klimatförändringar och hårdare regleringar av utsläpp av växthusgaser gör att det finns ett stort intresse för att minnna CO₂-emissionerna från dieselmotorer för tunga fordon. Termiska barriärskikt (TBC) kan användas för att isolera olika komponenter och därmed minska värmeöverföringarna från motorn, vilket kan leda till en förbättrad verkningsgrad för motorn. Den lägre temperaturen för isolerade komponenter kan även leda att deras livslängd ökar. TBC har även haft en begränsad framgång i dieselmotorer, beroende på problem med livslängd för beläggningen, lägre minskningar av värmeöverföringarna än väntat samt förändrade förbränningsegenskaper. I denna avhandling utvärderas livslängd och termiska egenskaper för termiska barriärskikt under förhållanden liknande de i en dieselmotor för tunga fordon, med det övergripande målet att minska värmeöverföringarna från motorn.


En stor inverkan av mikrostrukturen kunde även påvisas för plasmasprutad YSZ beträffande värmeledning och reflektans, utvärderad för temperaturer och våglängder för värmestrålning som är typiska för dieselmotorn. Beläggningarna med bäst egenskaper för förbränningsrummet var en nanostrukturerad beläggning, följd av en plasmasprutad beläggning med konventionell mikrostruktur. Dessa kombinerade låg värmeledning med hög reflektans vid temperaturer upp till 600°C. Tester med tunna metalliska ytskikt kunde inte ytterligare förbättra reflektansen för YSZ.

In situ-mätningar av värmeflödet från förbränningsrummet för plasmasprutade beläggningar visade att Gd₂Zr₂O₇ (GZ) var det material som hade bäst isolerförmåga, medan YSZ med olika sorter mikrostruktur samt La₂Zr₂O₇ gav endast små minskningar av värmeflödet. YSZ med porerna tätade med aluminiumfosfat gav ökat värmeflöde jämfört med en stålreferens. Långtidsprov visade ett positivt inkörningsbeteende för YSZ och GZ. Värmeflödet minskade för dessa beläggningar under de 2-3 första timmarnas prov, innan det stabiliserades.

Motorprov med TBC på kolvar visade bäst resultat för en suspensionsplasmasprutad (SPS) beläggning med GZ. Beläggningen minskade temperaturen på oljan i kolven och ökade temperaturen i avgaserna, men ökade inte motorns verkningsgrad.
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Anders Thibblin
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<th>Description</th>
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<tbody>
<tr>
<td>AHRR</td>
<td>Apparent heat release rate</td>
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<tr>
<td>APS</td>
<td>Atmospheric plasma spraying</td>
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<tr>
<td>ATDC</td>
<td>After top dead centre</td>
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<tr>
<td>CA</td>
<td>Crank angle</td>
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<tr>
<td>CaSZ</td>
<td>Calcia-stabilized zirconia</td>
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<tr>
<td>EB-PVD</td>
<td>Electron beam–physical vapour deposition</td>
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<tr>
<td>DHR</td>
<td>Directional hemispherical reflectance</td>
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<tr>
<td>GZ</td>
<td>Gadolinium zirconate</td>
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<tr>
<td>ISFC</td>
<td>Indicated specific fuel consumption</td>
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<tr>
<td>LHR</td>
<td>Low heat rejection</td>
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<tr>
<td>LZ</td>
<td>Lanthanum zirconate</td>
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<tr>
<td>PS-PVD</td>
<td>Plasma spray–physical vapour deposition</td>
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<tr>
<td>RQ</td>
<td>Research question</td>
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<tr>
<td>RT</td>
<td>Room temperature</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscope</td>
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<tr>
<td>SPS</td>
<td>Suspension plasma spraying</td>
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<tr>
<td>TBC</td>
<td>Thermal barrier coating</td>
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<tr>
<td>TCF</td>
<td>Thermal cycling fatigue</td>
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<tr>
<td>TGO</td>
<td>Thermally grown oxide</td>
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<tr>
<td>YSZ</td>
<td>Yttria-stabilized zirconia</td>
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List of appended publications

Paper A
The author was involved in planning the experiments and evaluating the results, and wrote parts of the paper.

Paper B
The author formulated the research questions and chose the methodology for answering them, designed the test rig and planned the experimental tests, supervised the experimental engine testing work, performed isothermal oxidation tests, performed some of the microstructural analyses, and wrote the entire paper.

Paper C
The author formulated the research questions and chose the methodology for answering them, planned the experimental tests, performed all microstructural analyses, porosity measurements, and Vickers hardness measurements, made the synthesis, evaluated the results of the remaining experiments, and wrote the entire paper.

Paper D
The author planned the tests, supervised the design of the heat flux probe, supervised the experimental engine testing work, performed the porosity measurements and most of the microstructural analyses, and wrote the entire paper.

Paper E
The author formulated the research questions and chose the methodology for answering them, planned the experimental tests and evaluated the results of the engine test, performed all microstructural analyses, and wrote the entire paper.
1. Introduction

Growing concern about climate change has increased the interest in techniques and regulations for reducing CO₂ emissions. Within the EU, heavy-duty vehicles account for approximately 5% of the total greenhouse gas emissions [1]. In 2019, the European Parliament called for a fleet average CO₂ emission reduction for new heavy-duty trucks of 15% by 2025 and 30% by 2030 relative to the 2019 level [1]. In the US, there are already regulations for reducing greenhouse gas emissions from heavy-duty vehicles [2]. CO₂ emissions from future heavy-duty vehicles need to be reduced to achieve these legislated reduction targets and reduce the impact on global warming. This means that, for heavy-duty vehicle powertrains, much effort needs to be put into developing electric drive and improved engine efficiency.

Part of the total CO₂ emission reduction could be obtained by using electric powertrains, for example, in distribution trucks that run relatively short distances every day or city buses that may have time to charge their batteries during stops. However, it is more difficult to use electric powertrains in long-haulage trucks, as very large and heavy battery packs would be required with today’s technology. Another potential challenge for the transformation to electric powertrains is the availability of materials for electric machines and batteries [3]. These are some factors indicating that for many years to come there will still be a need for internal combustion engines, powered with diesel or alternative fuels, for heavy-duty applications. To meet the CO₂ emission targets in EU legislation, it is therefore essential to further improve diesel engine efficiency.

Modern heavy-duty diesel engines can convert approximately 45% of the fuel energy into useful work. The rest of the fuel energy is lost, mainly as heat to coolant and exhaust (Fig. 1). If the heat transfer from the hot gases inside the combustion chamber to the metal walls could be decreased, the heat losses to the coolant and the oil could be reduced. According to the first law of thermodynamics, lower heat losses would mean that more energy is left in the gas to produce useful work on the piston and consequently improve the efficiency of the engine [4]. A method for achieving lower heat losses is to apply thermal barrier coatings (TBCs) to components such as pistons and valves.

![Figure 1: Illustration of efficiency and heat losses in an internal combustion engine](image)

Insulated combustion chamber components would offer several additional benefits, besides the potential to improve the engine efficiency. The heat loads on the components would be reduced, potentially improving their thermo–mechanical fatigue life. Oxidation would also be reduced, which also could prolong component life or permit operating conditions with higher gas temperatures. The reduced heat transfer into the components would imply that the size and capacity of the cooling system could be reduced, resulting in lower pumping losses as well as weight and cost reductions.

However, there has not yet been a major breakthrough in TBCs in diesel engines. Ever since the presentation in the 1970s of the low heat rejection (LHR) engine concept, with ceramic components or TBCs in the engines, numerous results of either improved or reduced engine efficiency have been reported. For heavy-duty diesel engines, some have reported 6% increased fuel efficiency [6] while others have reported 8% reduced efficiency [7].
The reasons for the varying results and why TBCs have not yet been a successful method for improving the efficiency of diesel engines are not yet fully understood. Several different factors that could be causing the sometimes poor TBC results have been proposed. Deteriorated combustion because of poor air and fuel mixing due to the rougher and hotter surface of the TBC is one possible factor [8]. Fuel becoming trapped in the pores and not being burnt in the early stages of the combustion cycle could also reduce the efficiency of the engine [8–10]. Higher-than-expected heat transfer to the TBC due to increased surface area [11] or transparency to thermal radiation [12] may also have an influence. These are some of many possible factors that may be resulting in the sometimes poor results.

There is consequently a gap in our knowledge of what is actually causing the often unexpectedly low efficiency of engines with TBCs. In the present work, lab tests are used to evaluate the thermal and optical properties of different TBCs. These results are then connected to the effectiveness of different coatings in reducing the heat flux from the combustion chamber. Furthermore, these results are then used when designing TBCs for pistons, to evaluate their impact on heat losses and engine efficiency.

TBCs can also be used for insulating the exhaust manifolds, where the coating is most easily applied to the outer manifold surface, as many coating techniques used to apply TBCs are line-of-sight processes. Insulated manifolds offer several different benefits. If coated with TBC on the outside, the heat losses from the manifolds to the surrounding air would be decreased. The under-hood temperature would be reduced and consequently also the heat load on surrounding components. This could prolong the lifetime of, for example, sensitive aluminium components or electronics. More energy would also be left in the exhaust, which could be recovered by the turbocharger or waste heat recovery systems. Exhaust after-treatment systems would also reach their light-off temperatures more quickly after cold starts, which may help reduce NOx emissions. However, by insulating the outer surface, the temperature of manifold materials, typically cast irons in heavy-duty diesel engines, would increase. Since the fatigue life of the manifold is sensitive to small changes in temperature [13], coating the outside may not be possible for all types of applications.

The same positive effects as obtained with external coating can be obtained by coating the inner surface of the manifolds, while the problems of shorter fatigue life can be avoided. TBC on the inner surface will instead reduce the cast iron temperature, potentially improving the fatigue life. The cast iron will also obtain improved oxidation resistance from the TBC, which is also beneficial for the lifetime of the component. However, there are limitations in what coating techniques can be used for coating the inner surface, as, for example, thermal spraying techniques are typically line-of-sight processes and cannot reach far into narrow pipes. Another potential problem is that if a TBC spalls off the inner surface, it may damage the turbocharger or other components. TBCs for the inner surface of manifolds therefore need to be tested thoroughly before being evaluated in a real engine, to avoid problems due to spallation. Evaluation methods for testing lifetime as well as thermal properties under conditions similar to those in a real engine are therefore required, and have been developed in this work.

1.1 Research objectives

To reduce the heat losses from the engine, the overall research objective is to evaluate TBCs with respect to lifetime and thermal insulation properties for conditions similar to those in a heavy-duty diesel engine.
1.2 Research questions

The research aims to answer the following general research question:

*How can thermal barrier coatings be designed for improved lifetime and insulation effectiveness for combustion chamber and exhaust components in a heavy-duty diesel engine?*

This main research question has been divided into the following more specific research questions (RQs):

RQ1: *How do different coating microstructures, produced using APS and PS-PVD, influence the thermal cycling lifetime of YSZ TBCs in low cycle fatigue tests? (Papers A–D)*

RQ2: *What is the thermal cycling performance of different TBCs like for exhaust manifold applications? (Papers A & B)*

RQ3: *How are different TBCs influenced by the thermal cycling fatigue exposure inside the combustion chamber? (Papers D & E)*

RQ4: *How is the substrate temperature of exhaust components influenced by different TBCs and different thicknesses of the coating? (Papers A & B)*

RQ5: *How are thermal insulation properties of TBCs influenced by different coating microstructures produced with APS, SPS, and PS-PVD, and by the addition of thin surface layers? (Papers C–E)*

RQ6: *How is the heat flux from the combustion chamber influenced by TBCs with respect to their composition, microstructure, and sealed porosity? (Papers D & E)*

RQ7: *Is there a running-in period, with respect to thermal insulation properties, for TBCs inside the combustion chamber? (Paper D)*

RQ8: *How are the efficiency and heat losses of an engine influenced by different types of TBCs produced by APS and SPS? (Paper E)*

1.3 Thesis outline

Chapter 1 gives an introduction to TBCs and the importance of research in this area. Chapter 2 describes the different layers of a TBC, the coating processes and materials that can be used, and important material properties for a TBC. Chapter 3 presents the challenges that TBCs are subjected to in a heavy-duty diesel engine as well as positive and negative effects of TBCs on engine performance. Chapter 4 describes the experimental methods used in answering the RQs and the TBC materials evaluated in the different papers. Chapter 5 presents the results. Chapter 6 discusses the results, followed by the conclusions and proposed future work.

1.4 Limitations

This research evaluates the thermal cycling lifetimes and thermal properties of TBCs for diesel engines, using existing test methods as well as in situ test methods developed in this work. The evaluation of how different TBCs influence the performance of a heavy-duty diesel engine is limited to one engine test with pistons coated with TBCs, which were selected based on promising properties and the results of earlier experiments. The influence of TBCs on emissions was not considered in this work. The temperature swing concept, of increasing interest for diesel engines in recent years, was not considered in this thesis.
2. Thermal barrier coatings

A typical TBC system for diesel engines is shown in Fig. 2. The first layer is the substrate material, which for diesel engine components is often steel, cast iron, or aluminium. The next layer, the bond coat, has two important functions: it should protect the substrate from oxidation and also provide sufficient bonding, often by having an irregular surface that promotes the mechanical bonding of the next layer, the top coat [14, 15]. The top coat, normally consisting of ceramic material, is a low-thermal-conductivity layer that provides the thermal insulation. The thermal conductivity of the top coat can be reduced by various microstructural modifications that reduce the phonon mean free path. The coating processes and materials used for the different layers will be further described in the following sections.

![Figure 2: Typical plasma-sprayed TBC system with a YSZ top coat, metallic bond coat (FeCrAlY), and cast iron substrate (SiMo51).](image)

2.1 Substrate material

The substrate material on which the TBC is applied can be of several different types in heavy-duty diesel engines. Pistons are commonly made of steel. The cylinder head is typically made of cast iron, with either lamellar or compacted graphite. Exhaust manifolds are often made of nodular cast iron, with additions of silicon and molybdenum to improve the thermo-mechanical fatigue and oxidation properties. Cast steel can also be used for these components. The substrate materials are thus much different from the superalloys that are typical substrate materials for TBCs in aircraft and land-based gas turbines [16], on which much TBC research concentrates. Typical TBC materials have a lower thermal expansion mismatch with steels and cast irons than with superalloys, which is a positive factor for their thermal cycling fatigue life. SiMo51, a nodular cast iron often used for manifolds, as well as piston steel both have a thermal expansion of approximately $12–13 \times 10^{-6} \text{ K}^{-1}$ [17, 18]. However, the oxidation resistance is lower for steels and cast irons, which leads to other challenges.

2.2 Bond coat material

For steel and cast iron substrates, bond coats are usually thermally sprayed alloys of type MCrAlX, where M stands for Fe, Ni, Co, or a combination of these elements, and X stands for oxygen-active elements, commonly Y but could also include Ta, Si, Hf, or other elements [16].

One purpose of the bond coat is to provide sufficient bonding to the top coat [14, 15]. However, because oxygen can easily pass through many top coats (e.g., the commonly used YSZ top coat) at high temperatures, an oxygen barrier is needed to protect the substrate. The bond coats are therefore designed to form a protective $\alpha\text{-Al}_2\text{O}_3$ layer in the interface between the bond coat and the
top coat [19]. Alumina grows very slowly at high temperatures due to its low oxygen diffusivity. The alumina layer is often referred to as thermally grown oxide (TGO). At lower temperatures, it has been shown that instead of alumina, a chromia layer may form for MCrAlY bond coats [20].

Yet another important role of the bond coat is to reduce stresses in the top coat. A ductile bond coat can reduce crack growth near the bond coat/top coat interface [21].

It has been suggested that the lower temperatures in diesel engines than, for example, gas turbines, may allow the use of a TBC without any bond coat [14] or with a bond coat having lower oxidation resistance. FeCrAlY alloys have been suggested as possible bond coats in diesel engines [22].

### 2.3 Top coat materials

A top coat material to be used in a diesel engine must fulfil a number of requirements [14, 23, 24]:

- low thermal conductivity
- low specific heat
- low density
- thermal expansion close to that of steel and cast iron
- strain compliance, because thermal expansion mismatch between the layers of a TBC system causes large strains during thermal cycling
- thermodynamic compatibility with the oxides that form in the interface between the top coat and the bond coat
- high flexural strength
- high fracture toughness
- high thermal shock resistance

The top coat can consist of a single layer or of several layers to combine the desired properties of different materials. For example, a first layer that provides toughness during thermal cycling could be combined with a second layer that protects the TBC from the infiltration of calcium-magnesium-alumino-silicates [25]. TBCs can also have graded structures, as they can effectively reduce thermal stresses in the coating [26]. Top coats with layered structures, with a first layer having good fracture toughness and a second layer having good thermal properties, have also been evaluated for some applications [25, 27, 28].

Some top coat materials with interesting properties for diesel engines will be discussed in this section.

#### 2.3.1 YSZ

The ceramic most often used as a top coat in aero and industrial turbines, and in diesel engine tests, is yttria partially stabilized zirconia (YSZ). Pure zirconia, ZrO$_2$, possesses many of the required properties listed above, but undergoes detrimental tetragonal-to-monoclinic phase transformation during cooling. This is associated with an increase in volume of approximately 3–5% [29, 30], which may result in coating failure. Zirconia is therefore normally stabilized with 6–8 wt% yttria (Y$_2$O$_3$). With these yttria contents, YSZ coatings have compositions placing them in a two-phase region of the phase diagram, where yttrium-lean tetragonal and yttrium-rich cubic phases are found (see Fig. 3). However, due to rapid cooling during the coating processes, a metastable t´ phase is formed instead of the two-phase mixture. When subjected to high temperatures exceeding 1200°C during long-term operation, the t´ phase is decomposed through diffusion into a two-phase mixture of the tetragonal and cubic phases [15, 30]. This may cause spallation of the coating due to volume change when the tetragonal phase later transforms to the monoclinic phase. However, the t´ phase can be expected to be stable at the relatively low temperatures in a diesel engine.
Additions of yttria or other stabilizers result in a significant number of vacancies, which are effective in scattering phonons and reduce the thermal conductivity of the material [32]. The type of deposition process also greatly influences the thermal conductivity of the coating. For 6–8 wt% YSZ, the thermal conductivity is typically 1.8–2.0 Wm⁻¹K⁻¹ when deposited by electron beam–physical vapour deposition (EB-PVD) and can be as low as 0.5–1.0 Wm⁻¹K⁻¹ for plasma-sprayed coatings [19]. The coefficient of thermal expansion is $10.7 \times 10^{-6}$ K⁻¹ for plasma-sprayed YSZ with 7.8 wt% Y₂O₃ in the 30–1000°C temperature interval [33]. This is close to the thermal expansion of steel and cast iron. A close thermal expansion match is important in reducing the stresses during thermal cycling.

2.3.2 Gd₂Zr₂O₇
Gadolinium zirconate (GZ, Gd₂Zr₂O₇) is a ceramic with a pyrochlore structure. Gadolinium zirconate has excellent thermal stability and can be used at temperatures above 1300°C [34]. The thermal conductivity of a plasma-sprayed GZ coating is about 20% lower than that of plasma-sprayed YSZ [35]. The lower thermal conductivity is related to the higher number of oxygen vacancies (contributing to phonon scattering) and to the large difference in atomic weight between Gd and Zr in GZ, that is also contributing to phonon scattering [36, 37]. GZ has also been shown to have a higher reflectance than YSZ, which is due to its higher refractive index (2.9 for GZ versus 2.1 for YSZ) [38]. The large difference in refractive index from that of air gives GZ a high scattering coefficient and high reflectance. The thermal expansion of GZ is $10.4 \times 10^{-6}$ K⁻¹ at 30–1000°C, which is similar to that of YSZ [39].

It has been demonstrated that GZ reacts with alumina in the TGO to form GdAlO₃, which may compromise the oxidation protection provided by the TGO [40]. To prevent this reaction, it is recommended that a diffusion barrier, such as YSZ, be used or that the temperature near the TGO be kept well below 1100°C [40]. Because bond coat temperatures in diesel engines in general are much lower than 1100°C, this might not be a problem for such applications.

2.3.3 La₂Zr₂O₇
Lanthanum zirconate (LZ, La₂Zr₂O₇) is another ceramic with a pyrochlore structure. Its fracture toughness is comparable to that of YSZ and its thermal conductivity is about 20% lower than that of
YSZ at elevated temperatures [33]. Other properties that make lanthanum zirconate interesting as a top coat is its low sintering tendency and high-temperature stability up to 2000°C [33, 34].

The thermal expansion of lanthanum zirconate is $9.1 \times 10^{-6}$ K$^{-1}$ at 30–1000°C, which is lower than that of YSZ and may lead to higher stresses in the coating due to a larger thermal expansion mismatch with other layers in the TBC system [35, 34]. However, the results of thermal cycling tests of LZ and YSZ/LZ coatings have been promising [33, 34].

When materials are being plasma sprayed, constituents can be lost. In the case of La$_2$Zr$_2$O$_7$, La$_2$O$_3$ easily evaporates, which may lead to monoclinic and cubic ZrO$_2$ in the coating [41, 42]. As was described in the section about YSZ, pure ZrO$_2$ may undergo phase transformations at elevated temperatures, which may cause spallation of the coating. At high temperatures (1400°C), LZ has been observed to react with Al$_2$O$_3$, which exists in the TGO layer [41]. However, no reaction was seen at lower temperatures, so this is not expected to be a problem in diesel engine applications. Ionic conductivity is much lower for LZ than for YSZ [41], which means that oxygen is not as easily transported though the top coat, and the bond coat oxidation rate can be expected to be lower.

### 2.3.4 Mullite

Some top coat materials that have insufficient temperature stability for turbine applications may still be of interest for diesel engines, due to their lower temperatures. One such material is mullite (3Al$_2$O$_3$·2SiO$_2$). It has been demonstrated that plasma-sprayed mullite contains amorphous phases that crystallize and decrease in volume at temperatures of 750–1000°C [43, 44]. This volume contraction, along with a low thermal expansion ($4.5 \times 10^{-6}$ K$^{-1}$ between room temperature (RT) and 1400°C [45]), may lead to cracks in the top coat and to spallation in high-temperature applications [43, 46, 47]. However, some tests in diesel engines have shown improved lifetime of mullite compared with YSZ [7, 48].

However, problems of spallation and increased thermal conductivity due to crystallization during high-temperature exposure can be avoided in diesel engines, where coating temperatures are fairly low. Lab tests as well as thermal cycling tests of pistons have indicated that mullite top coats last longer than do zirconia top coats [7, 48, 49]. The crystallization also increases the thermal conductivity of mullite. The RT thermal conductivity of a plasma-sprayed coating increases from about 1.3 to 1.8 Wm$^{-1}$ K$^{-1}$ after one heating cycle to 1000°C [50].

### 2.3.5 Forsterite

Forsterite (MgO·2SiO2) is a top coat material with potential for a good thermal cycling lifetime in diesel engines. This is because its high thermal expansion coefficient of $11.0 \times 10^{-6}$ K$^{-1}$ between 25 and 800°C [51] makes it a good match for steels and cast irons. The small thermal expansion mismatch between forsterite and 80Ni-20Cr bond coat with steel substrate has led to good results in thermal cycling tests [51]. Thermal conductivity decreases with temperature, declining from about 1.8 Wm$^{-1}$ K$^{-1}$ at RT to 1.3 Wm$^{-1}$ K$^{-1}$ at 1000°C [50].

### 2.4 Coating processes and microstructures

Several different coating processes can be used to deposit TBCs on metallic substrates. Some of these coating processes, and typical microstructural features associated with them, are described below.

#### 2.4.1 APS

Atmospheric plasma spraying (APS) is widely used due to its high deposition rate, relatively low cost, ability to coat large components, and high-porosity microstructure that reduces the thermal conductivity [52].

In APS, particles with a diameter of typically 5–50 µm are injected into a plasma jet with a temperature typically in the range of 8000–14,000 K [53, 54]. The particles are heated and accelerated towards the substrate by the plasma jet. On impact, the molten particles flatten and form splats (see Fig. 4). As more splats hit the surface, a lamellar coating is built up. The high
cooling rates during the solidification of the splats leads to directional solidification and grain growth, mainly perpendicular to the substrate [53, 55].

The microstructure of APS coatings can be modified by changing, for example, the feedstock composition and morphology, plasma gas composition and gas flow, and substrate temperature [52, 53]. Three types of APS microstructures – conventional, nanostructured, and segmented – will be discussed below.

2.4.2 APS, conventional

Plasma-sprayed TBCs with conventional microstructure typically have 10–15% porosity [56]. The porosity consists of various types of pores and cracks (see Fig. 5). Large pores are formed when splats do not completely fill the holes between previously sprayed particles [19, 52, 57]. Interlamellar pores and cracks are caused by factors such as shadowing by previously deposited particles and splat-curling caused by surface tension and thermal expansion mismatch stresses [57]. Intrasplat cracks are caused by stresses from the rapid cooling of the splats, from the cooling of the entire coating, and from solid-state phase transformations [58]. The porosity provides some lateral strain compliance for APS coatings with conventional microstructure [14].

2.4.3 APS, nanostructured

There are limitations to how small feedstock particles can be for use in an APS process, due to problems injecting light particles into the viscous flame [59]. This problem can be avoided by using nanostructured, agglomerated feedstock particles. A microstructure comprising semi-molten particles surrounded by fully molten particles (see Fig. 6) can then be built up with atmospheric
plasma spraying [60]. This type of microstructure, with nanozones embedded in a conventional microstructure, is often referred to as a “nanostructured” or “bimodal” microstructure [60].

Improved thermal cycling lifetime [61–63] and decreased thermal conductivity [61, 64] compared with those of conventional microstructure have been reported. The better thermal cycling lifetime can be explained by lower residual stress as well as a lower elastic modulus [62]. The low thermal conductivity of nanostructured coatings can be attributed to their higher porosity, mainly due to the increased amount of microporosity [61], and increased phonon scattering due to the increased extent of grain boundaries [65, 66] and increased submicron porosity [65]. The reduced thermal conductivity due to decreased grain size is most noticeable at low temperatures, while this effect is insignificant at temperatures exceeding 1000°C [67]. The mean free path of the phonons decreases with temperature, until it is limited by the interatomic spacing. The grain boundaries therefore do not limit the phonon mean free path unless the grain boundaries are opaque to phonon transmission. For tetragonal zirconia, which is not opaque to phonon transmission across the grain boundaries, the thermal conductivity is not grain-size dependent at temperatures above 1000°C [67]. However, at the significantly lower temperatures in a diesel engine, grain size may be an important factor reducing the thermal conductivity of the top coat.

Figure 6: Nanostructured APS coating, showing nanozones consisting of semi-molten agglomerated particles surrounded by splats formed by fully-molten particles.

2.4.4 APS, segmented microstructure

Segmentation cracks are vertical cracks that run perpendicular from the surface (see Fig. 7) to a depth of at least 50% of the top coat thickness [68]. TBCs with such segmentation cracks can be manufactured using the APS process by adjusting the spray parameters. As described in section 2.3.1, the solidification rate is very high when plasma spraying ceramics, which leads to directional solidification from the substrate towards the surface. If the coating surface is kept at a relatively high temperature, increased diffusion will improve the bonding between the splats, increase the grain size, and promote the growth of microcracks over several splats [53]. A high feeding rate or low relative speed between the spray gun and the coated specimen can be used to obtain greater pass thickness, which also promotes the growth of larger grains and vertical cracks [53].

Exposure of conventional TBCs to high temperatures leads to tensile stresses in the top coat due to thermal expansion mismatch between the different coating layers, followed by stress relaxation [53]. The vertical cracks in the segmented coating allow the top coat to expand and contract in a lateral direction during thermal cycling, which reduces the tensile stresses in the top coat during the high-temperature exposure [53, 68]. This gives lower stress relaxation during high-temperature exposure and consequently lower compressive strains in the coating during the ensuing cooling stage [53]. Thermal cycling tests have demonstrated that the improved strain tolerance due to the segmentation cracks gives a longer coating lifetime than that given by conventional APS microstructure [53, 68, 69].
2.4.5 EB-PVD

Electron beam–physical vapour deposition (EB-PVD) coatings have a columnar microstructure with inter-columnar gaps that permit lateral strain compliance similar to that of the segmented APS coating [14]. EB-PVD coatings are therefore often used on mechanically loaded parts in aero and industrial turbines. The part to be coated is preheated and inserted into a vacuum chamber, where electron beams melt and evaporate ceramic ingots. The vapour, mixed with a controlled amount of oxygen, is deposited on the substrate [32]. This is an expensive coating process and, due to high deposition temperatures of about 1000°C [70], cannot be used with many of the alloys used for diesel engine components.

2.4.6 PS-PVD

Plasma spray–physical vapour deposition (PS-PVD) is a relatively new process that combines some of the main advantages of both the APS and EB-PVD processes. A columnar microstructure, similar to that of EB-PVD coatings, can be produced by the PS-PVD process [71] (see Fig. 8). This type of microstructure, with featherlike columns separated by gaps, provides a high strain tolerance [71, 72]. Because it is a plasma spray process, it is lower in cost and has a higher deposition rate than the EB-PVD process [73].

The spraying distance in the PS-PVD process can be more than 2 m and the plasma plume can be 0.4 m wide [54]. The long spraying distance, low pressure (about 0.1 mbar) in the PS-PVD chamber, and high power of the plasma gun allow evaporation of the injected powder [72, 73]. When the vapour reaches the surface, a columnar structure layer starts to grow. Crystals of certain orientations grow faster and create columns, while the slower-growing crystals stop growing when they reach another column [74]. Gaps between the columns are then created, leading to the high strain tolerance of the coating.

For PS-PVD coatings, the thermal conductivity is reportedly about 0.8–1.1 Wm⁻¹ K⁻¹ [72, 73]. These low values are explained by the high porosity and large number of defects in the microstructure [72, 73]. The coating process requires a high substrate temperature, normally about 900–1100°C [72], which limits its use for diesel engine components.
2.4.7 SPS
Suspension plasma spraying (SPS) is a relatively novel coating process that combines several of the interesting properties of APS, PS-PVD, and EB-PVD. The feedstock powder is suspended in a liquid, usually water or ethanol [19]. The use of a suspension allows the use of much smaller particles than in an APS process, i.e., particles in the nanometre or sub-micron size range [75]. The suspension is then fed into the plasma jet and accelerated towards the target. During the flight, the suspension is atomized into fine droplets, followed by evaporation of the liquid, agglomeration of the particles, and melting [36, 59]. The smaller particles than those in APS means that the particles are flattened less on impact. The small particles may also partially follow the gas flow near the surface, which can lead to shadowing effects and the growth of a columnar structure [36, 76] (Fig. 9). Different types of microstructures can be obtained with the SPS process, including lamellar, columnar, and vertically cracked structures [36].

It has been shown that the thermal conductivity of YSZ coatings produced by SPS can be as low as 0.5–0.9 Wm\(^{-1}\) K\(^{-1}\) due to the high amount of microporosity in the coating [77, 78]. Another interesting property of SPS coatings is their high hemispherical reflectance, compared with that of APS, which may be related to the high porosity and higher amount of microporosity in the SPS coating [79].

Figure 9: YSZ coating fabricated using the SPS process.

2.4.8 Sol-gel and slurry coatings
Slurry spraying is a relatively cheap and simple method for producing TBCs [80]. The slurry, which consists of powder suspended in a fluid, is applied to a surface in several layers using a spray gun. After drying, the multilayer coating is pressed in a compression chamber, followed by sintering in a
furnace or with an acetylene torch [80]. A great advantage of this coating method is the ability to coat complex geometries, including surfaces inside pipes that are out of direct sight [80]. Slurry coatings can also be applied by dipping the substrate in a mixture of ceramic powder and then leaving it to dry. This is repeated until the desired thickness has been achieved, after which the coating is densified by hot isostatic pressing.

The sol-gel process is another non-line-of-sight process. First, a solution typically consisting of metal alkoxides in an organic solvent is formed. A sol-gel coating can be manufactured by spraying the solution or dipping the substrate in it, followed by gravitational draining and evaporation of the solvent [81]. The dried sol is then compacted in a firing process into a hard oxide coating. A sol-gel coating with incorporated ceramic particles is shown in Fig. 10.

Figure 10: Sol-gel composite TBC, showing CaSZ (large particles) and YSZ (small particles) in a silica matrix.

2.5 Thermal properties
The thermal conductivity of a TBC is governed by the intrinsic thermal conductivity of the top coat material and by the microstructural features, such as cracks and pores.

2.5.1 Intrinsic thermal conductivity
The thermal conductivity, $k$, of insulating solids can be expressed as:

$$k = \frac{1}{3} C v l$$

(1)

where $C$ is the heat capacity per unit volume, $v$ is the phonon velocity, and $l$ is the mean free path of the phonons [29, 82]. According to the Dulong–Petit law, $C$ reaches a constant value of $3k_B$ at temperatures above the Debye temperature, where $k_B$ is the Boltzmann’s constant. This gives:

$$k = k_B v l$$

(2)

The minimum thermal conductivity, $K_{\text{min}}$, of the solid can then be calculated [29, 82]. The velocity of the sound in the material can be used as an approximate value of the mean phonon velocity, $v_{\text{min}}$:

$$v_{\text{min}} \approx 0.87 \sqrt{E/\rho}$$

(3)

where $E$ is the elastic modulus and $\rho$ is the density. The dimension of the molecule determines the minimum phonon mean free path, $l_{\text{min}}$, according to:

$$l_{\text{min}} = \left( \frac{N_A m_p}{M} \right)^{\frac{2}{3}}$$

(4)
where \( N_A \) is Avogadro’s number, \( M \) is the molar mass, and \( m \) is the number of atoms per molecule. Combining equations 2–4 then gives:

\[
k_{\text{min}} = 0.87 k_B \left( \frac{N_A m \rho}{M} \right)^{2/3} \left( \frac{E}{\rho} \right)^{1/2}
\]  

From Eq. 5 it can be seen that the minimum thermal conductivity can be altered in various ways [29]. Adding alloying elements will influence the molar mass and density, and therefore change the minimum thermal conductivity of the solid. Using novel oxide ceramics will influence the molar mass, number of atoms per molecule, density, and also the elastic modulus. The calculated and experimental minimum thermal conductivities of various TBC materials are shown in Fig. 11.

![Figure 11: Minimum thermal conductivity values of TBC materials and other ceramics [83].](image)

### 2.5.2 Influence of microstructure

The thermal conductivity of a solid can be reduced by various modifications of the microstructure. Introducing high porosity and microcracks oriented perpendicular to the heat flux, as is the case for the interlamellar cracks in an APS coating, are effective ways to reduce the thermal conductivity [84, 85]. Microcracks has been shown to be more dominant than the porosity for reducing the thermal conductivity [86, 87]. Two important features of the microcracks are their orientation and the presence of islands of partial contact along the microcracks [86, 87]. Thermal radiation also contributes to the heat transport through a semi-transparent ceramic. Imperfections in the ceramic, such as grain boundaries and pores of the same dimensions as the wavelength of the thermal radiation, increase the photon scattering and can thus reduce the radiative heat transfer [12, 88].
3. Insulated engines

The potential benefits of TBCs inside the combustion chamber and manifolds, as well as some problems related to TBCs, were presented in chapter 1. In this chapter, some issues related to TBCs in a heavy-duty diesel engine are further discussed.

TBCs are exposed to a harsh environment in a diesel engine, where they must withstand the temperature variations from the combustion (see Figs. 12 and 13), the start/stop of the engine, high pressures, and a corrosive environment. The high temperature of the flame means that the TBCs must not only have low thermal conductivity but also optical properties that reduce the radiative heat transport through the coating.

The introduction of TBCs in a diesel engine may influence factors such as the heat release rate, volumetric efficiency, and emissions. These factors are introduced in this chapter to give an overview of some of the problems that may be encountered in an insulated engine.

Figure 12: Schematic view of diesel engine showing the combustion chamber and manifolds, where TBCs can be applied.

Figure 13: Surface temperatures of combustion chambers with zirconia coatings of different thicknesses [89].
3.1 Durability of TBCs for manifolds and combustion chambers

3.1.1 Durability

TBC durability is an important issue. Heavy-duty trucks often need to run 1–2 million km before being taken out of use. If TBCs fail before that, the engine may be severely damaged. Thermal protection of the substrate is lost, which can cause problems with oxidation, thermal fatigue, and fractured components. A spalled TBC can cause problems for the turbocharger, as large flakes of TBC can damage the turbine wheel blades. Particles from a spalled TBC may also cause excessive wear of the cylinder walls and, if bypassing the piston rings to the oil sump, wear of bearings and other components.

Spallation is generally caused by high stresses in the bond coat/top coat interface, causing crack initiation and growth [32]. These stresses are caused by, for example, TGO growth, differences in thermal expansion between different layers, and mechanical load. These and other factors, such as sintering and phase transformations, that may cause spallation problems will be discussed below.

3.1.1.1 Stresses from the coating process

The coating process and thermal cycling conditions during operation generate stresses in the coating; these stresses may cause crack initiation and growth, and eventually coating failure.

Quenching stresses arise in APS coatings during the thermal spraying process. The impacted particles solidify rapidly, but their contraction is restricted by adherence to the underlying coating layers. As a result, tensile quenching stresses form in the coating [19]. Thermal expansion mismatch between the coating and substrate causes stresses in the coating during cooling after spraying. These thermal mismatch stresses in the coating are typically compressive, because the ceramic usually has a lower thermal expansion than does the metallic substrate and therefore shrinks less [19].

3.1.1.2 Thermal stresses

Thermal expansion mismatch between the different layers in the TBC system causes stresses in the coating during thermal cycling. The ceramic top coat has a lower thermal expansion than does the metallic substrate, which results in stresses at the top coat/bond coat interface. The magnitude of the stresses tends to increase with each successive cycle, and may eventually result in coating failure [90]. The TGO growth also results in stresses at the top coat/bond coat interface, which may be a contributing factor for coating failure.

3.1.1.3 Sintering and densification

When plasma-sprayed coatings are exposed to high temperatures, the interlamellar cracks can change shape and become spheroidized [14]. This will cause an increase in thermal conductivity over time [14], and there is also a risk of spallation as the densification will produce a volume decrease in the top coat [15]. Another aspect to consider is the decrease in strain compliance. Porous coatings can have good strain compliance due to the low elastic modulus associated with high porosity [10, 14]; consequently, densification will lead to decreased strain compliance.

Because densification is temperature dependent, the service temperature and the thermal conductivity of the coating are important factors to consider. Low thermal conductivity will result in large temperature gradients within the coating and, if the service temperature is high enough, large density gradients as well due to densification [14]. The part of the TBC where densification is most likely to occur is the outermost top coat closest to the high-temperature gas.

The increase in thermal conductivity due to sintering can be significant at elevated temperatures [91, 92]. The sintering of microcracks at the splat interfaces together with decreased porosity have been identified as the main reasons for the large increase in the thermal conductivity of coatings after high-temperature exposure [29, 92, 93].

The aging effects discussed above are most noticeable with high-temperature exposure, such as in aircraft- and land-based gas turbines. Exposure within diesel engines has also been shown to
influence the effectiveness of the coating over time. Heat flux measurement within a light-duty diesel engine indicated increasing heat flux due to coating degradation [94].

### 3.1.1.4 Phase stability/transformations

High-temperature exposure can cause the crystallization of amorphous phases or the phase transformation of top coats, as described in section 2.3. This often leads to a volume change, as in the tetragonal-to-monoclinic phase transformation of YSZ, which causes a volume increase of 3–5% [29]. A volume change often leads to a risk of failure due to the formation of cracks in the TBC. At the moderate temperatures in diesel engines, YSZ should not undergo phase transformations; however, if other materials are used as the top coat, this factor needs to be considered.

### 3.1.1.5 Bond coat oxidation

Oxygen is easily transported through YSZ at elevated temperatures and reacts with the bond coat. Preferably, a slow-growing α-alumina layer, TGO, is formed in the bond coat/top coat interface. The oxidation of the bond coat is associated with growth stress.

Evans [95] has analysed the stresses caused by a fast-growing TGO and noted that large out-of-plane tensile stresses form on the flanks of protuberances in the bond coat/top coat interface; Evans also noted that these stresses are likely to cause the nucleation of cracks, while strain energy in the top coat contributes to the final failure. For aircraft engines, it has been found that the critical TGO thickness is typically 6–7 µm [29].

### 3.2 Influence on combustion characteristics

The theoretical increase in fuel efficiency from applying TBCs to combustion chamber components is often difficult to achieve in practice. Some potential problems associated with TBCs in the combustion chamber are a lower heat release rate, fuel entrapment in pores, increased convective heat transfer, and decreased volumetric efficiency. These issues will be discussed below.

#### 3.2.1 Volumetric efficiency

Volumetric efficiency is defined as the amount of fresh air trapped in the combustion chamber divided by the theoretical maximum amount of air in the cylinder [96]. TBCs may have a negative effect on the volumetric efficiency, because the intake air is heated by the hotter walls of an insulated combustion chamber and because of the higher residual gas temperature than in a standard engine [97]. The density of the intake air is decreased and less air can enter the combustion chamber, meaning that the volumetric efficiency drops [97, 98]. Increased turbocharging can be used to maintain high volumetric efficiency [97, 98].

#### 3.2.2 Heat release rate

Reduced fuel efficiency has been reported from several tests of engines with thermal insulation, which has been explained by a lower heat release rate [9, 97, 99, 100]. The low heat release rate may be due to several factors. Fuel and gas may be trapped in the porous TBC resulting in less fuel being burnt during the first part of the combustion cycle, shifting the heat release towards the later part of the cycle and giving less useful work on the piston [8–10, 101, 102]. Also, several researchers have attributed the lower heat release rate to the rough surface of the TBC, which has a negative effect on the fuel and air mixing and reduces the motion of the flame [94, 103]. An example of the shift in heat release is shown in Fig. 14.
3.2.3 Fuel and air entrapment in pores

The ceramic top coat is porous, which allows air and fuel to penetrate into the coating, especially during the combustion stroke when the pressure can exceed 200 bars in the combustion chamber. Entrapment of fuel in the coating would lead to worsened fuel efficiency, as less fuel may be combusted in the early stage of the combustion stroke. Hot air pushed into the pores would increase the transfer of heat to the coating [94]. To minimize the effect of fuel and air entrapment, a sealing coating can be used. In the case of a light-duty diesel engine, it has been shown that the insulation is improved by using a sealing layer on top of the thermal barrier, the effect being attributed to reduced intrusion of gas into the coating [94].

There are several methods to produce a sealing layer: a dense layer can be applied by thermal spraying onto the ceramic top coat [25]; porosity can be sealed by penetrating dip processes; or an organometallic coating can be applied by chemical vapour deposition processes [10]. Perhydropolysilazane, which reacts with water vapour and forms silica, has been used for sealing porous alumina coating [94]. Aluminium phosphate sealing and laser glazing have also been used for sealing zirconia-based TBCs [105]. Yet another method is to apply a film of phosphate glass on the TBC surface and cure it at elevated temperature [10].

3.2.4 Increased convective heat transfer coefficient

In some tests, insulated walls have actually appeared to increase the heat losses from the combustion chamber. In tests of an insulated piston in a diesel engine, Woschni et al. observed increased heat transfer at higher temperatures [106]. They proposed that the boundary layers were thinner for the hot TBC surfaces, leading to a drastic increase in the convective heat transfer coefficient. However, later investigations have instead attributed these problems to degraded combustion [89].

TBCs in the as-coated condition typically have much rougher surfaces than do uncoated steel pistons, which could be another reason for lower fuel efficiency than expected. For steel pistons, it has been demonstrated that a polished surface improves the fuel efficiency, as the heat transfer area is reduced [107]. Tests of TBCs with varying degrees of surface roughness have found increased heat transfer with rougher surfaces [108]. When testing a metallic TBC on the piston of a spark-ignition (SI) engine, Marr et al. [109] found that the average heat flux was higher than for an uncoated surface, which was attributed to the greater surface roughness. Memme et al. [110, 111] demonstrated that, in an SI engine, a smooth TBC on the piston results in higher gas temperatures and pressures, and a 3% improvement in fuel efficiency compared with the same coating in as-sprayed condition. Somhorst et al. have shown that the surface roughness of a TBC on the piston of a light-duty diesel engine has a significant effect on heat losses as well as on the fuel.
Because an increased heat transfer coefficient counteracts other benefits that can be obtained by using TBCs, surface roughness seems to be an important issue.

3.2.5 Emissions
The higher surface temperatures in an insulated engine influence the amount of emissions. The formation of nitrogen oxides, NOₓ, is highly temperature dependent. Diesel engines with TBCs could therefore be expected to produce more NOₓ, as has been shown in many experimental studies [113–115]. However, some researchers have measured lower NOₓ emissions, attributing this to, for example, decreased premixed combustion [97].

Hydrocarbon emissions are expected to decrease in an insulated engine due to a smaller quenching distance near the walls and also because of an increased lean flammability limit [23, 97]. The oxidation reactions are helped by the higher temperatures in the combustion chamber, with less unburned hydrocarbon as a result [113, 114]. This has been confirmed by most experimental studies [97, 114, 115].

3.3 Thermal radiation
YSZ, which is the most common TBC top coat material, is partially transparent to thermal radiation (see Figs. 15 and 16) [12, 116]. The thermal conductivity of TBCs typically decreases with temperature, but thermal radiation is proportional to T³ [91]. At high temperatures, transparency to thermal radiation may thus be an important factor to consider. The bond coat or substrate can be directly heated by external radiation if a translucent TBC is used [12]. Also, thermal radiation generated within the top coat will transport heat from the hotter outer parts of the coat near the surface towards the substrate [12]. For gas turbines, a theoretical model of the heat flux has shown that the contribution from thermal radiation becomes significant at temperatures above 400°C [91].

![Figure 15](image-url)

**Figure 15:** The optical scattering and absorption coefficients of YSZ as well as black-body radiation at several temperatures. The absorption coefficient is low at most wavelengths generated in an engine. Much of the radiation that is not reflected is therefore transmitted through the coating [14].
Thermal radiation is a more important aspect for TBCs on combustion chamber components than on exhaust components. This is due to thermal radiation from the hot flame and to greater temperature differences between the gas and the bond coat as well as between the top coat surface and the bond coat. Thermal insulation of the TBC inside the combustion chamber is therefore dependent on the thermal conductivity as well as optical properties of the TBC. The importance of the optical properties inside the combustion chamber also varies between engines, due to factors such as the amount of exhaust gas recirculated into the engine, compression ratio, load, and type of fuel. An illustration of the energy balance of a TBC in a diesel engine is shown in Fig. 17.

There are large variations in the estimates and measurements of how much thermal radiation contributes to the total heat losses to the combustion chamber walls in uninsulated diesel engines. Skeen et al. [117] have shown that, in some engines, the thermal radiation energy coming from soot equals approximately 0.5% of the fuel energy, while Borman et al. [118] have shown that thermal radiation may account for 20–40% of the total in-cylinder heat flux.

A highly reflective top coat would reduce the problems with external radiation heating the substrate. It has been demonstrated that adding a thin, reflective aluminium film on top of YSZ reduces the temperature beneath the TBC [99]. In turbine applications, it has been argued that a highly reflective surface layer would be the most effective way to reduce the radiative heat transfer, but that it would be difficult for such a layer to withstand the erosion in that application [14]. Metallic coatings on the surface or embedded in the ceramic may also lead to problems, due to thermal expansion mismatch or poor bonding between the metal and the ceramic [120]. Others have successfully improved the reflectance by coating with multiple layers of YSZ and Al₂O₃ using EB-PVD [121].

Instead of increasing the reflectance, attempts have been made to increase the absorbance of TBCs by incorporating NiO into the coating [14]. However, those attempts were not very successful as they caused an increase in both the thermal conductivity and sintering rate.

Modifying the microstructure can also improve the reflectance. Imperfections, such as microcracks and pores, increase the scattering within the coating, resulting in less radiation transmitted to the substrate and consequently a higher reflectance [120, 122].

Figure 16: Reflectance and transmittance of GZ and YSZ coatings [119].
Figure 17: Physical model of the energy balance of a TBC in a diesel engine. The TBC is exposed to heat flux from thermal radiation of short wavelength from the combustion, $q_0$, and of long wavelength from the hot gas, $q_1$; it also experiences convective heat transfer from the gas, $q_{\text{conv}}$. Thermal radiation is reflected by the surface, $q_s$, reflected within the coating, $q_r$, absorbed within the coating, $q_a$, and transmitted, $q_t$, to the substrate. There is also a conductive heat flux within the coating, $q_{\text{cond}}$, and a radiative heat flux from the hot TBC surface, $q_E$ (adapted from Merzlikin et al. [123]).
4. Methodology

In this chapter the methodology used for answering the RQs is presented. The methodology is divided into three parts:

1. Thermal cycling lifetime testing
2. Thermal and optical property testing
3. Engine testing with insulated pistons

Parts 1) and 2) were initially conducted on a small scale, with lab tests for a small coin-shaped specimen (Fig. 18). The results of those initial tests were used in later stages when selecting TBCs for further tests and when designing large-scale in situ tests in engines. Results from the first two parts were used when selecting TBCs, for evaluating their impact on engine performance when applied to pistons, i.e. part 3 of the methodology. A summary of the different coatings evaluated using all the different test methods is presented in Table 1. YSZ with conventional microstructure, applied by APS, is used as a reference in all the tests. This reference coating was produced with some different spray guns and bond coats in the different papers, as shown in Table 2. The reference had despite the different spray guns relatively similar porosity in Papers B-E, while the reference coating in Paper A had lower porosity. Nanostructured YSZ APS showed slightly higher variation in porosity and fraction of nanozones (Table 2).

Table 1: Summary of TBCs and evaluation methods (TCF = thermal cycling fatigue). The letters A-E denote the different papers where results from these tests are presented.

<table>
<thead>
<tr>
<th>Top coat material</th>
<th>Coating process</th>
<th>Top coat microstructure (+ surface modification)</th>
<th>Test methods used in the different papers</th>
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<td>TCF in furnace</td>
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<td>Sol-gel (dipped)</td>
<td>Dense</td>
<td>A</td>
</tr>
<tr>
<td>GZ</td>
<td>APS</td>
<td>Conventional</td>
<td>A</td>
</tr>
<tr>
<td></td>
<td>SPS</td>
<td>Conventional</td>
<td></td>
</tr>
<tr>
<td>LZ</td>
<td>APS</td>
<td>Conventional</td>
<td>A</td>
</tr>
<tr>
<td>Mulilte</td>
<td>APS</td>
<td>Conventional</td>
<td>A</td>
</tr>
<tr>
<td>Forsterite</td>
<td>APS</td>
<td>Conventional</td>
<td>A</td>
</tr>
<tr>
<td>Only bond coat</td>
<td>APS</td>
<td>Conventional</td>
<td>A</td>
</tr>
</tbody>
</table>

*) Fewer interlamellar cracks than are normally seen in a conventional type of microstructure.
Fig. 18: Test samples. Upper left: part of a coated piston, upper centre: heat flux probe, upper right: coated pipe, lower left: sample for thermal cycling tests, lower centre: sample for thermal cycling tests, thermal conductivity measurements, and optical measurements, lower right: sample for thermal cycling tests inside the manifold.

Table 2: Manufacturing conditions and porosity of the conventional APS YSZ and nanostructured APS YSZ.

<table>
<thead>
<tr>
<th>Bond coat material</th>
<th>Conventional APS YSZ</th>
<th>Nanostructured APS YSZ</th>
<th>Spray gun for top coats</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Feedstock material</td>
<td>Feedstock material</td>
<td>Feedstock material</td>
</tr>
<tr>
<td>Paper A</td>
<td>Amdry 962</td>
<td>Amdry 204 NS-1</td>
<td>Metco 222 A</td>
</tr>
<tr>
<td>Paper B</td>
<td>Amdry 9700</td>
<td>Amdry 204 NS-1</td>
<td>Metco 222 A</td>
</tr>
<tr>
<td>Paper C</td>
<td>Amdry 9700</td>
<td>Amdry 204 NS-1</td>
<td>Metco 222 A</td>
</tr>
<tr>
<td>Paper D</td>
<td>Amdry 9700</td>
<td>Amdry 204 NS-1</td>
<td>Metco 222 A</td>
</tr>
<tr>
<td>Paper E</td>
<td>Amdry 386</td>
<td>204 B-NS</td>
<td>-</td>
</tr>
</tbody>
</table>

Fig. 19 is an illustration of the TBCs that have been evaluated in the different papers. In Paper A, several different top coat materials, produced with APS, were investigated, together with two sol-gel coatings. YSZ was identified as an interesting material to study further for diesel engine applications, while the sol-gel coatings had too high thermal conductivity.

Then, in Paper C, APS YSZ coatings with different types of microstructures were investigated. PS-PVD YSZ was also included, while SPS was not included due to problems finding a supplier. Three APS YSZ coatings (conventional, nanostructured and segmented) were selected for the next paper, while PS-PVD was excluded from further studies, despite having interesting properties, because of high costs and problems to coat e.g. cast iron and steel due to the high process temperature.

In Paper D it was investigated how some APS YSZ coatings and also GZ and LZ produced with APS perform inside the combustion chamber. Conventional YSZ and GZ were identified as two of the most interesting TBCs for further studies inside the combustion chamber.
In Paper E, SPS coatings were evaluated for pistons. YSZ and GZ were selected as top coat materials, based on the results in Paper D. APS YSZ was included as a reference.

Paper B describes a thermal cyclic test rig that was developed, and how a few TBCs are influenced by the thermal cyclic exposure. Conventional and nanostructured APS YSZ were selected for this study based on the results in Papers A and C.

Figure 19: Illustration of the coatings and coating processes that have been selected for the different papers.

4.1 Thermal cycling fatigue

The long lifetime of a TBC is of great importance in heavy-duty diesel engines as the vehicles are often expected to run for 1–2 million km. Several different methods for comparing the thermal cycling fatigue (TCF) life of TBCs have therefore been used in this work.

The TCF life of TBCs for aero engines or industrial gas turbines is often tested using either burner rig tests or cycling furnace tests. These tests usually have 1100–1200°C as their maximum temperature [78 124–126], which is much higher than what is reached in a diesel engine. Results reported from such tests can therefore not be directly applied in heavy-duty diesel engine applications. The limiting factor for the lifetime of TBCs in thermal cycling tests performed at such high temperatures is often the thickness of the TGO [29]. However, as TGO growth rates are much lower at typical diesel engine temperatures, other test conditions are required for evaluating the thermal cycling lifetime in such applications.

The test methods used in this work for evaluating TCF life are:
- two types of thermal cycling test in a furnace – with and without forced cooling
- three types of exposure to the cyclic conditions within a heavy-duty diesel engine:
  - exposure inside a manifold
  - exposure to exhaust in a test rig
  - exposure to cyclic conditions within the combustion chamber on two different types of components – heat flux probes and pistons

4.1.1 Furnace exposure

Two different furnace tests were used for evaluating the thermal cycling lifetimes of different TBCs, i.e., with and without forced cooling. The temperatures in these tests were chosen to simulate the low cycle fatigue a TBC would be exposed to in a component such as an exhaust manifold or turbo manifold.
4.1.1.1 Cyclic furnace test without forced cooling

Coin-shaped cast iron specimens with different TBCs were tested in a chamber furnace without any forced cooling during the cooling stage. The temperature was cycled between 180°C and 760°C, with a 15-min hold time at the maximum temperature. These temperatures were chosen to imitate the conditions in a gas exchange test cycle, which is a test cycle used for evaluating exhaust components. The duration of each cycle was 140 min. Some of the samples were tested for 500 h while the rest were tested for 1000 h. The temperature cycling profile is shown in Fig. 20.

Figure 20: Programmed temperature profile of the TCF test in a chamber furnace.

4.1.1.2 Cyclic furnace test with forced cooling

TCF tests using faster heating and cooling rates were conducted at Linköping University. Four TBC specimens at a time were positioned on a test table. During the heating stage, a furnace kept at 800°C was positioned over the specimens. The furnace was removed after 1 h, and the surface of the TBC specimens was cooled for 320–370 s with compressed air until the temperature reached 100°C. The heating and cooling procedure was repeated 500 times. When visual inspection revealed spallation covering 20% or more of the surface, the specimen was removed. Fig. 21 shows the furnace and the temperature profile for the first three cycles of a TCF test.

In TCF tests for aerospace and gas turbines, temperatures often reach 1100–1200°C and the growth rate of TGO is often a critical factor [124]. NiCoCrAlY, which has high oxidation resistance at high temperatures due to the formation of a stable alumina layer on the surface, is commonly used as the bond coat in aerospace and industrial turbine applications. The temperature (800°C) used in the TCF test was chosen as it is similar to the highest temperatures the gas and the material in the manifolds reach in the diesel engine. Exhaust gas temperatures and consequently the materials in the manifolds do not typically exceed 760°C [13]. The temperature of the bond coat is much lower for many other components, especially the ones subject to forced cooling, such as the pistons and the cylinder heads. These lower temperatures also justify using bond coats with lower oxidation resistance, so FeCrAlY was used for some of the studied TBCs. Consequently, to imitate the conditions in a diesel engine and to avoid too-rapid TGO growth, the test temperatures need to be rather low.
4.1.2 Engine exposure

Furnace exposure is a common method for evaluating the thermal cycling lifetimes of TBCs, but does not include all the mechanisms that can lead to failure in a diesel engine. Some parameters that could influence the results but are missing in most furnace tests are the gas pressure variations, correct gas composition, and correct temperature gradient through the samples. If actual components coated with TBC were instead tested in an engine to evaluate the thermal cycling lifetime, it would be necessary to run several costly engine tests to evaluate several candidate materials. Also, considering the relatively large TBC area of a coated manifold, there is a large risk of damaging the turbocharger or other engine components if the TBC spalls off. Due to the high cost of running multiple long-term engine tests as well as the risk of damaging the engine, there is a need to develop alternative test methods for evaluating the thermal cycling lifetimes of different TBCs in their correct environments before selecting certain TBCs for further tests in an engine. Two different test methods for evaluating TBCs for manifolds have therefore been developed, and the effects of these tests on different TBCs have been evaluated. TBCs exposed to the engine conditions inside the combustion chamber were also analysed with respect to TCF damage.

4.1.2.1 Exposure inside exhaust manifold

The first test method for evaluating the thermal cycling lifetimes of TBCs for manifolds uses specimens shaped as screws, coated with TBC (see Fig. 22). The specimens were inserted into threaded holes (see Fig. 22), specially made for this purpose, to expose the TBCs to the exhaust gases. The coatings were then tested for 2000 h in a thermal cycling engine test (i.e., gas exchange test cycle) in which the exhaust gas reached temperatures up to 760°C (see Fig. 23). The TBCs could not be inspected during the test, but were analysed when removed after the full 2000-h test.

One advantage of this method compared with testing in a furnace is that the coatings are exposed to the correct temperature, gas, and pressure conditions. A disadvantage is the limited number of samples that can be tested simultaneously, as the lifetime of the manifolds may be reduced if too many holes are made in them. Advantages compared with testing actual components having the entire inner surface coated with TBC include a lower risk of damaging the turbocharger if the coating spalls off (due to the smaller area being coated) and faster and less expensive testing if multiple coatings are to be evaluated.
4.1.2.2 Thermal cycling in exhaust test rig

The second test method developed was a test rig connected to a heavy-duty diesel engine. This was developed to allow more samples to be tested simultaneously and to allow testing of samples having a more realistic geometry. The specimens were shaped as short pipes with the inner surface coated with TBC (Fig. 24). One advantage of this geometry compared with the screw-shaped specimen is that the coating will have a microstructure that is more representative of what is obtained in a manifold, as, for example, an APS coating produced with a steep spraying angle will be more porous than an APS coating sprayed perpendicularly on a flat sample [129]. Also, the stresses within the specimens deriving from gas pressure and temperature cycling will be more similar to the real conditions of a coated manifold.

A Scania DC13 heavy-duty diesel engine with 12.7 dm³ displacement volume was used for the test. An exhaust manifold was modified with an extra outlet to allow part of the exhaust flow to be directed to a test rig where several coated pipes were positioned in a series (Fig. 25). An axial force was applied, as well as sealing rings between the pipes, preventing gas from escaping between the pipes. The exhaust from the engine was led through the pipes, alternately heating and cooling them from the inside. A gas restrictor was positioned in the exhaust pipe after the test rig, ensuring that the turbocharger received sufficient gas flow. No exhaust from the test rig was led back to the engine, to prevent any damage to the engine if the TBCs were to spall off.

The engine was run constantly at 750 rpm. Each thermal cycle consisted first of a heating stage at 70% engine load for approximately 35 min, followed by 15 min at 0% load. The gas temperature within the coated pipes was approximately 530°C at the end of the heating stage and 50°C at the end of the cooling stage (Fig. 26). The test was run for 150 cycles before the coatings were inspected for failures.
Thermocouples were positioned within the gas stream inside the coated pipes to monitor the exhaust gas temperature during the test. Encapsulated thermocouples were also positioned on the outer surface of the pipes to measure the surface temperature trends. This was done to evaluate whether it was possible to measure the effectiveness of different TBCs or detect spallation or aging effects, such as sintering or deposit formation on the inside.

Three different coatings were tested in the rig (Fig. 27). Two of these were TBCs with YSZ applied by APS as the top coat material. One of these had a conventional type of microstructure, while the second one was a nanostructured TBC, i.e., it had a bimodal structure consisting of nanostructured grains embedded in a conventional microstructure. One oxidation-resistant coating, NiCoCrAlY applied by APS, was also evaluated. This type of coating could potentially reduce the growth of oxide scale on the inner surface by preventing oxygen from reaching the cast iron, thereby improving the lifetime of the component, but the temperature reduction of the cast iron would be less than if a TBC were used.

Figure 24: Specimens with TBC on the inner surface.

Figure 25: Thermal cycling test rig for TBCs for manifolds.
4.1.2.3 Thermal cycling exposure inside the combustion chamber

The TCF to which TBCs are subjected differs between the combustion chamber and the exhaust manifold. In the combustion chamber, there is a combination of high cycle fatigue and low cycle fatigue. The heating of the surfaces during the combustion and the subsequent cooling of the surfaces during the following cycles cause high cycle fatigue in the coatings. The slower temperature variations due to factors such as the start/stop of the engine and variations in load give rise to low cycle fatigue. The thermal cycling tests of the type described in section 4.1.1 could be used as accelerated tests of the low cycle fatigue life of TBCs to be used inside combustion chambers as well as for manifolds. However, it is more difficult to design an accelerated high cycle fatigue test for conditions similar to the ones inside the combustion chamber, due to the fast cycling (typically 10-15 Hz). In this work, TBCs tested on heat flux probes (section 4.2.3) or on pistons (section 4.3) were analyzed after exposure inside the combustion chamber to evaluate whether there was any damage from the combined low/high cycle fatigue conditions. The longest time for the high cycle fatigue exposure was 5 h at 1200 rpm, which corresponds to 180,000 cycles.

4.2 Insulating properties

4.2.1 Thermal conductivity

The thermal diffusivity of the coatings was measured using laser flash analysis equipment (LFA427; Netzsch, Selb, Germany) at Jönköping University. Steel specimens with a diameter of 12.8 mm and a thickness of approximately 1.5 mm, coated with TBC on one of the circular faces, were used for the laser flash measurements. A thin layer of graphite was applied to the coated sample to maximize the amount of thermal energy absorbed by the surface during measurements. The thermal diffusivity
measurements were performed at temperatures ranging from RT up to 800°C. The analysis software used the Netzsch method for three-layer systems, i.e., the 3L heat loss + pulse correction model, to calculate the thermal diffusivity of each layer.

In laser flash measurements, the coated side of the tested sample is heated by a pulse of energy from a laser and the temperature response on the reverse of the sample is measured. Thermal diffusivity, \( \alpha \), can then be determined as:

\[
\alpha = 0.1388 \frac{L}{t(0.5)}
\]

where \( L \) is the thickness of the sample and \( t(0.5) \) is the time needed for the reverse of the sample to reach 50% of its maximum temperature [130]. The thermal conductivity, \( k \), is calculated as:

\[
k = \alpha \cdot C_p \cdot \rho
\]

where \( C_p \) is the specific heat capacity and \( \rho \) is the density. Literature data were used for the specific heat capacity, while the density was calculated as:

\[
\rho = (1 - f) \cdot \rho_0
\]

where \( f \) is the fraction of pores in the coating and \( \rho_0 \) is the density of a pore-free specimen.

### 4.2.2 Optical properties

Reflectance measurements were performed at temperatures ranging from 20 to 700°C, using a Vertex 70 FTIR spectrometer (Bruker, Billerica, MA, USA).

The directional hemispherical reflectance (DHR) at RT was measured using an integrated sphere at wavelengths of 1.25–33 µm. With the high-temperature, high-pressure cell used for measurements at elevated temperatures, DHR cannot be measured directly. Instead, the spectral specular reflectance, \( R(T) \), was measured at temperatures ranging from 350 to 750°C in 50°C steps, as well as at RT, \( R(RT) \). The DHR at high temperatures could then be calculated according to:

\[
DHR(T) = DHR(RT) \cdot \frac{R(T)}{R(RT)}
\]

### 4.2.3 In situ heat flux measurements for the combustion chamber

A heat flux probe (see Fig. 28) that can be inserted into the combustion chamber of a heavy-duty diesel engine was developed in two master’s thesis projects [131, 132]. One intake valve was removed from a single-cylinder 2.1 dm³ Scania diesel engine and was replaced with a stationary heat flux probe. The engine was thus running with only one intake valve and two exhaust valves working. The still functioning intake valve and valve seat were adjusted and tested in a gas flow test rig to ensure that the swirl number was the same as when two intake valves were being used.

The main parts of the heat flux probe are: (i) a support ring that is fastened in the position of the valve seat; (ii) a semi-hollow shaft that is internally cooled with water or compressed air, has four thermocouples at different heights, and has a circular face where TBCs can be applied and exposed to gases in the combustion chamber; and (iii) a retainer that pushes the shaft against the support ring to prevent the leakage of combustion gases into the intake port. If the thermal conductivity, \( k \), of the steel in the shaft is known, the heat flux through the TBC and up through the shaft of cross-sectional area \( A_s \) can be calculated by measuring the temperature, \( T_1 \) and \( T_2 \), at different heights with distance \( L \) between them. The heat flux in the vertical direction, \( q_z \), can then be calculated using Fourier’s law:

\[
q_z = -\frac{k \cdot A_s \cdot (T_2 - T_1)}{L}
\]
Figure 28: A heat flux measurement probe positioned in one of the intake valves of a single-cylinder diesel engine. The coating to be tested is applied to the lower surface of the shaft (adapted from Kianzad [132]).

Efforts were made to ensure that the heat flux was as one-dimensional as possible, i.e., that most of the heat flux through the tested TBC went up through the shaft and as little heat flux as possible went horizontally to/from the support ring or into the air surrounding the shaft within the intake port. Internal cooling of the shaft improved the heat flux in the vertical direction. Water was used for internal cooling in short-term tests to maximize the temperature gradient in the vertical direction. Long-term running-in tests used compressed air instead of water as the cooling medium, which enabled the probe to reach higher temperatures and the aging of the TBC to be further accelerated.

Thermocouples in the support ring were used in evaluating the temperature difference between the shaft and support ring at a certain distance from the combustion chamber. Insulating coatings of different thicknesses were tested on the lower face of the support ring. This was done to adjust the heat flux into the ring and, consequently, the temperature of the ring. The coating thickness that minimized the temperature difference between the ring and the shaft was used for all the following tests when the heat flux through the shaft with different TBCs was measured.

The contact area between the shaft and ring was made conical, with slightly different angles for the two components to ensure that the contact area was small and situated at the lower end of the shaft. A small contact area minimized the potential heat flux between the two components, and a contact area near the combustion chamber prevented hot gases from leaking into the contact and heating the shaft from the side.

A copper washer was used to prevent leakage between the support ring and cylinder head. Gas leakage in that position would heat the shaft from the side and cause measurement errors. The intake port was blocked to prevent intake gas from entering the intake port and cooling the shaft from the side.

The size of the coated area of the shaft was intentionally made relatively small (135 mm²). This area was considered small enough so that any effects of the different coatings on the combustion characteristics could be neglected.

The single-cylinder engine used for the test had a displacement of 2.1 dm³. During the measurements, it operated at a constant 1200 rpm with 120 mg of fuel/injection. Engine data and test parameters are shown in Table 3.
Table 3: Engine data and test parameters.

<table>
<thead>
<tr>
<th></th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Displacement</td>
<td>$2.123 \text{ dm}^3$</td>
</tr>
<tr>
<td>No. of cylinders</td>
<td>1</td>
</tr>
<tr>
<td>Engine speed</td>
<td>1200 rpm</td>
</tr>
<tr>
<td>Bore/stroke</td>
<td>130/160 mm</td>
</tr>
<tr>
<td>Compression ratio</td>
<td>18.5:1</td>
</tr>
<tr>
<td>Load, short-term test</td>
<td>$120 \pm 0.6 \text{ mg/injection}$</td>
</tr>
<tr>
<td>Load, 5-h running-in test</td>
<td>$120 \pm 1.0 \text{ mg/injection}$</td>
</tr>
<tr>
<td>Boost pressure</td>
<td>0.98 bar</td>
</tr>
<tr>
<td>Exhaust pressure</td>
<td>0.98 bar</td>
</tr>
<tr>
<td>Swirl number</td>
<td>1.7</td>
</tr>
<tr>
<td>Rail pressure</td>
<td>1200 bar</td>
</tr>
<tr>
<td>Start of injection</td>
<td>$-3^\circ$</td>
</tr>
<tr>
<td>Test duration for short-term test</td>
<td>$3 \times 15 \text{ min}$</td>
</tr>
<tr>
<td>Internal cooling of shaft for short-term test</td>
<td>Water $24.8 \pm 1.1^\circ \text{C}, 0.90 \text{l/min}$</td>
</tr>
<tr>
<td>Test duration for running-in test</td>
<td>$1 \times 5 \text{ h}$</td>
</tr>
<tr>
<td>Internal cooling of shaft for running-in test</td>
<td>Compressed air: 7.7 bar pressure</td>
</tr>
</tbody>
</table>

4.2.4 Thermal modelling

One of the main benefits of TBCs on the inner surface of manifolds is the reduced cast iron temperature, which may improve the lifetime of the component. Temperature distribution in the pipe under stationary conditions was calculated to be better able to estimate the benefits of different TBCs.

To calculate the temperature distributions, equations for a straight cylindrical pipe with TBC on the inner surface, presented by Bejan et al., were used [133]. First, the heat transfer per unit length, $q$, is calculated as:

$$ q = \frac{T_{\text{in}} - T_{\text{out}}}{2\pi H_{\text{in}}a + \frac{\ln((\alpha + h)/a)}{2\pi k_{\text{TBC}}} + \frac{\ln(c/(a + h))}{2\pi k_{\text{Iron}}} + \frac{1}{2\pi H_{\text{out}}c}} $$

where $T_{\text{in}}$ is the exhaust temperature, $T_{\text{out}}$ is the ambient temperature, $a$ is the inner radius, $c$ is the outer radius, $h$ is the coating thickness, $k_{\text{TBC}}$ is the thermal conductivity of the TBC, $k_{\text{Iron}}$ is the thermal conductivity of the cast iron, $H_{\text{in}}$ is the heat transfer coefficient on the inside, and $H_{\text{out}}$ is the heat transfer coefficient on the outside. The temperature on the outer surface, $T_{\text{c}}$, of the pipes can then be calculated as:

$$ T_{\text{c}} = T_{\text{out}} + \frac{q}{2\pi H_{\text{out}}c} $$

4.3 Engine testing of coated pistons

An engine test of the pistons coated with TBC was conducted in a single-cylinder test cell. The purpose of the test was to determine how pistons with SPS TBCs influence the temperature of the piston cooling oil, cylinder head temperature, exhaust temperature, and indicated specific fuel consumption, compared with a piston with conventional APS TBC and an uncoated steel piston.

Coating the entire combustion chamber could have been an option for studying the effects of TBCs on the engine’s heat losses and performance. However, to minimize the complexity, cost and work it might be better to study only one coated component at a time. The piston has been selected in this study based on some different factors. The heat losses to the piston are about equal to the heat losses through the cylinder head. However, the flame has a stronger interaction with the piston surface, as it is directed towards the piston bowl. Therefore, by choosing the piston to be coated, effects from the flame/TBC interaction, such as fuel being trapped inside the coating, will be included in the measurement results.
4.3.1 Temperature measurements

A simplified view of the test and the positions of the thermocouples is shown in Fig. 29. An oil cup was positioned below the piston to collect the cooling oil leaving the piston cooling galleries. The temperature of the oil in the cup was measured with a type-K thermocouple. By comparing the cooling oil temperatures for the different pistons, the effectiveness of different TBCs in reducing the heat losses through the piston can be evaluated.

A type-K thermocouple was also positioned within the cast iron of the cylinder head, close to the combustion chamber. The insulated pistons influence the temperature of the gases within the combustion chamber, and may also influence the combustion characteristics, which could result in more heat transfer to components such as the cylinder head and valves. The thermocouple in the cylinder head was used to measure such effects.

When insulating combustion chamber components, the exhaust temperatures often increase [97]. A type-K thermocouple was therefore positioned in the exhaust manifold to measure the exhaust temperature.

The same set of thermocouples was used for all the piston tests. The error, calculated as one standard deviation from the measurement data, was 0.95°C for the exhaust temperature, 0.27°C for the piston cooling oil temperature, and 0.46°C for the cylinder head temperature.

![Figure 29: Illustration of the test bench showing the positions of the thermocouples for measuring the oil, exhaust, and cylinder head temperatures.](image)

4.3.2 ISFC and AHRR

The indicated specific fuel consumption (ISFC) was calculated for the tests of different pistons as:

$$ ISFC = \frac{BSFC \cdot BMEP}{IMEP} $$

(13)

where $IMEP$ is the net indicated mean effective pressure, $BMEP$ is the brake mean effective pressure, and $BSFC$ is the brake specific fuel consumption.

The pressure in the combustion chamber was measured and used in calculating the apparent heat release rate (AHRR), which can be calculated as [134]:

```
\[ AHRR = \frac{\gamma}{\gamma-1} p \frac{dV}{dt} + \frac{1}{\gamma-1} V \frac{dp}{dt} \]  \hspace{1cm} (14)

where \( \gamma \) is the ratio of specific heats (\( \frac{C_p}{C_v} \)), \( p \) is the pressure, and \( V \) is the volume. The AHRR can then be used in determining the crank angle (CA) at which a certain fraction of the fuel has been burnt, commonly 10\%, 50\%, and 90\%. This can then be used when analysing what effect a TBC has on the combustion characteristics.

4.4 Material characterization

4.4.1 Microscopy
An Axio light optical microscope (Zeiss, Oberkochen, Germany) and a Sigma VP scanning electron microscope (SEM) (Zeiss) were used to acquire micrographs of the coatings. INCA software for energy-dispersive X-ray analysis (Oxford Instruments, Abingdon, UK) was used to analyse the chemical composition of oxides formed within the TBCs. AxioVision (Zeiss) and ImageJ image analysis software (National Institutes of Health and University of Wisconsin, USA) were used to measure porosity from the SEM images.

4.4.2 Surface measurements
A PLµ 2300 optical imaging profiler (Sensofar, Terrassa, Spain) was used to acquire the surface profiles of TBCs, followed by the evaluation of roughness parameters using Sensomap 4.1 (Sensofar) software. Surface roughness measurements were also made using a BFW A 10-45-2/90 roughness probe (Mahr, Göttingen, Germany) with a 2-\( \mu \)m radius of the stylus tip.
5. Summary of results

In this chapter, the results of the experiments are summarized. The results are divided into those concerning TCF, insulating effectiveness, and the engine testing of coated pistons.

5.1 Thermal cycling fatigue

5.1.1 Furnace exposure

5.1.1.1 Cyclic furnace test without forced cooling
Thermal cycling fatigue testing of TBCs inside a chamber furnace indicated the best results for the APS nanostructured YSZ. No damage from the thermal cycling could be observed. The APS YSZ with conventional microstructure displayed increased microcracking in the top coat. These results support previous results [61, 62] showing improved strain tolerance and lifetime of nanostructured YSZ compared with conventional YSZ.

The other three APS TBCs displayed damage of varying degrees from the TCF test. The LZ TBC displayed surface cracks and an increased number of interlamellar cracks. The surface cracks led to the spallation of micrometre-sized flakes after 1000 h of thermal cycling in the furnace. The mullite TBC displayed lateral cracks in the top coat/bond coat interface after furnace testing as well as some spallation at the edges. The forsterite TBC had lateral cracks near the bond coat/top coat interface after the TCF test. Fig. 30 shows typical examples of cracks and spallation found in APS coatings.

Figure 30: SEM images of the coating microstructure after thermal cycling in air for 1000 h showing (a) the mullite coating and (b) the forsterite coating. The increased amount of interlamellar cracks in LZ after 1000 h (d) can be seen when compared to LZ before the test (c), (from Paper A).
The two sol-gel composite coatings had an increased number of transversal cracks but no signs of spallation. Transversal cracks are generally good for the strain tolerance and lifetime of TBCs [53, 69], so the increased crack density was not considered to be a problem for this type of coating.

Oxidation of the substrate under the bond coat was observed in all the plasma-sprayed TBCs. The non-uniform oxidation and increase in volume can be expected to cause strains within the TBC and have a negative influence on the lifetime of the coatings.

5.1.1.2 Cyclic furnace test with forced cooling

The cyclic test in a furnace with forced cooling produced large differences in lifetime for the different YSZ TBCs (Figs. 31 and 32). APS-coated specimens with nanostructured and conventional microstructures all failed before 85 cycles. APS-coated specimens with segmented microstructure lasted much longer, i.e., 350–450 cycles, due to the higher strain tolerance provided by the vertical cracks. The FeCrAlY bond coat in these TBCs was severely oxidized (see Fig. 33). The severe oxidation shows that the bond coat temperature must be lower than the temperature during the TCF test (800°C) for FeCrAlY to be a viable alternative. The APS coatings partly failed in both the TGO and YSZ layers (Fig. 33).

The PS-PVD specimens with a NiCoCrAlY bond coat were run-outs without any signs of spallation after 500 cycles. The NiCoCrAlY bond coat was used instead of FeCrAlY for the PS-PVD specimens due to the high substrate temperature during the coating process. The combined effects of a more oxidation-resistant bond coat, a thinner top coat, and high strain tolerance due to the columnar microstructure explain the excellent results. The TGO thickness was only about 1.5 µm.

![Figure 31](image-url): Thermal cycling results for four different YSZ coatings. Coating failure occurred in the interval between the blue and red triangles (from Paper C).
Figure 32: Specimens after thermal cycling. The entire top coat has spalled off the three APS YSZ coatings, which show fractures partly in the TGO and partly in the YSZ top coat. The PS-PVD YSZ coating has survived the 500 cycles (adapted from Paper C).

Figure 33: SEM images of cross sections of samples after thermal cycling: (a) conventional APS YSZ with a 25-µm TGO layer, (b) nanostructured APS YSZ with a 30-µm TGO layer, (c) segmented APS YSZ with a 90-µm TGO layer, and (d) PS-PVD YSZ with a 1.5-µm TGO layer. The APS coatings failed partly in the TGO and partly in the YSZ, while the PS-PVD was intact after the test (from Paper C).
5.1.2 Engine exposure

5.1.2.1 Exposure inside exhaust manifolds
As in the furnace test, the nanostructured YSZ performed best in the TCF test inside manifolds, displaying no signs of damage after the exposure.

The other APS TBCs displayed similar results, regarding type and number of cracks, as in the furnace test. One difference was the amount of spallation for mullite: the entire top coat had spalled off from the mullite TBC during the TCF exposure inside the manifold.

Oxidation of the substrate under the bond coat (Fig. 34) was observed in all the plasma-sprayed TBCs after the thermal cycling inside the exhaust manifold, as in the furnace test. However, the composition of oxide scale was different, consisting of only Si oxide after the manifold exposure.

![Figure 34](image)

Figure 34: SEM images of conventional YSZ after thermal cycling in diesel exhaust gas for 2000 h: a) oxidation of the SiMo51 substrate and b) the oxidation at higher magnification (from Paper A).

5.1.2.2 Thermal cycling in exhaust test rig
The TCF performance of three different APS coatings was evaluated in the exhaust test rig. The two APS YSZ coatings, with nanostructure and conventional microstructure, displayed an increased number and size of cracks within the top coat after the thermal cycling (Fig. 35). This crack growth occurred even though the exhaust temperature in the test rig was lower than would be experienced in an exhaust manifold and the number of cycles was relatively low (150 cycles). The crack growth can likely be attributed to relatively poor intersplat bonding within the top coat. Coating the inner surface of a narrow pipe is difficult, as the particles will hit the wall at a small angle. This causes high porosity [129], as was seen in the two APS coatings, and poor intersplat bonding.

![Figure 35](image)

Figure 35: Conventional YSZ after the engine test showing oxidation of the SiMo51 substrate and cracks within the YSZ top coat (from Paper B).
Oxidation of the substrate beneath the bond coat was seen for the two TBCs and the APS NiCoCrAlY coatings that were also tested (Fig. 35). The bond coat appears to be too porous to protect the substrate from oxidation. No TGO was formed for these TBCs.

5.1.2.3 Thermal cycling exposure inside the combustion chamber

TBCs were exposed to thermal cycling conditions inside the combustion chamber when tested on pistons and on the heat flux probe. The coating microstructures associated with the highest strain tolerance, i.e., the SPS columnar and APS segmented microstructures, generally displayed the best results in these two types of cyclic exposure. The only damage to such coatings after engine testing was some small pits in an SPS YSZ top coat on a piston (Fig. 36a and b) and some cracks in a lateral direction at the lip of the piston with an SPS GZ top coat. Analysis of polished cross sections of the pits (Fig. 36b) revealed a horizontal band with high porosity below the pit and what appeared to be loosely bonded particles. The pits in the SPS YSZ top coat can therefore be explained by difficulties spraying a uniform coating with sufficient cohesive strength in parts of the piston bowl. The complex geometry causes turbulent flows, which result in debris with poor bonding on parts of the surface, as well as problems spraying a uniform coating due to shadowing effects. It could not be determined whether the lateral cracks in the SPS GZ top coat were formed in the coating process or in the engine test. Another type of microstructure shown to be more strain tolerant than the conventional type of microstructure is the nanostructure produced with APS [61, 62]. A nanostructured YSZ coating survived a short test on the heat flux probe without displaying any signs of damage.

![Image](image_url)

Figure 36: a) Small pits in the SPS YSZ top coat on a piston after the engine test (Paper E), b) cross section of the pits in the SPS YSZ top coat (Paper E), c) APS conventional YSZ after exposure in a heat flux probe (Paper D), and d) GZ after exposure in a heat flux probe (Paper D).

One of the APS YSZ TBCs with conventional microstructure, tested on a piston, also survived the test without any damage. The remaining APS TBCs with conventional microstructure displayed some cracks within the top coat after exposure on the heat flux probe. Conventional YSZ displayed
some growth of microcracks (Fig. 36c), while conventional LZ and GZ coatings (Fig. 36d) had larger and more severe cracks.

5.1.3 Summary, thermal cycling fatigue tests
A summary of the TCF life of the evaluated TBCs in the different TCF tests is presented in Table 4. The colour markings in the table show how the different evaluation methods and results are connected to RQs 1–3. Four of the TCF tests (in manifolds and furnaces) evaluated the low cycle fatigue behaviour of TBCs and are connected to RQs 1 and 2. The tests inside the combustion chamber mainly examine the influence of high cycle fatigue on the lifetime of TBCs and are connected to RQ 3. The connections between the results and the RQs are further discussed in chapter 6.

Table 4: Summary of the thermal cycling fatigue (TCF) test results in comparison with the reference, APS conventional YSZ. Grading: 0 similar to reference, ++ much better, + better, - worse, - - much worse than reference. Colour markings show the connections to RQ1, RQ2, and RQ3.

<table>
<thead>
<tr>
<th>Top coat material</th>
<th>Coating process</th>
<th>Top coat microstructure</th>
<th>Results of the different TCF tests</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>TCF inside manifold</td>
</tr>
<tr>
<td>YSZ</td>
<td>APS</td>
<td>Conventional</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>APS</td>
<td>Conventional sealed</td>
<td></td>
</tr>
<tr>
<td></td>
<td>APS</td>
<td>Nanostructured</td>
<td>+</td>
</tr>
<tr>
<td></td>
<td>APS</td>
<td>Segmented</td>
<td></td>
</tr>
<tr>
<td></td>
<td>PS-PVD</td>
<td>Columnar</td>
<td></td>
</tr>
<tr>
<td></td>
<td>SPS</td>
<td>Columnar</td>
<td></td>
</tr>
<tr>
<td>CaSZ/YSZ</td>
<td>Sol-gel (sprayed)</td>
<td>Dense</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Sol-gel (dipped)</td>
<td>Dense</td>
<td></td>
</tr>
<tr>
<td>GZ</td>
<td>APS</td>
<td>Conventional</td>
<td></td>
</tr>
<tr>
<td></td>
<td>SPS</td>
<td>Columnar</td>
<td></td>
</tr>
<tr>
<td>LZ</td>
<td>APS</td>
<td>Conventional</td>
<td></td>
</tr>
<tr>
<td></td>
<td>SPS</td>
<td>Columnar</td>
<td></td>
</tr>
<tr>
<td>Mullite</td>
<td>APS</td>
<td>Conventional</td>
<td></td>
</tr>
<tr>
<td>Forsterite</td>
<td>APS</td>
<td>Conventional</td>
<td></td>
</tr>
<tr>
<td>Only bond coat</td>
<td>APS</td>
<td>Conventional</td>
<td>0</td>
</tr>
</tbody>
</table>

*) Short test <1 h  
**) Suspected crack from sample preparation  
***) Damage from either coating process or engine exposure

5.2 Insulating properties
The effectiveness of a TBC is dependent on several factors. Low thermal conductivity and high hemispherical reflectance are two factors contributing to an effective TBC. The results of measurements of these properties for different TBCs are presented below.

Thermal conductivity results are used in calculating the insulating effects of different TBCs for exhaust manifolds under stationary conditions, a matter also presented in this section.

Some other factors affecting the effectiveness of a TBC are the surface roughness and open porosity of the top coat, as these may influence the heat transfer coefficient. To see the combined effect of all these factors, it is therefore important to measure the effectiveness of a TBC in situ. Such measurement results for the combustion chamber are presented below.
5.2.1 Thermal conductivity

Thermal conductivity results for the coatings from three of the papers are presented in Fig. 37. Interlamellar cracks and high porosity are microstructural features that are effective in reducing thermal conductivity [84]. The results indicate, as expected, high thermal conductivity for the TBCs with a small number of interlamellar cracks and low porosity. Mullite had almost no interlamellar cracks and consequently a relatively high thermal conductivity of 2.7 Wm\(^{-1}\) K\(^{-1}\). Sol-gel and segmented YSZ, which both have low porosity and a small number of interlamellar cracks, also have high thermal conductivity, 8.2 Wm\(^{-1}\) K\(^{-1}\).

Some of the TBCs with the lowest thermal conductivity were APS LZ (0.8 Wm\(^{-1}\) K\(^{-1}\)) and SPS GZ (0.62 Wm\(^{-1}\) K\(^{-1}\)). Both LZ and GZ have been shown by others to have lower thermal conductivity than YSZ [33, 35]. The different YSZ TBCs – i.e., conventional APS, nanostructured APS, and columnar SPS – showed no clear differences indicating which type of microstructure was the best. There were relatively large variations in the results for the same type of microstructure but produced on different occasions.

![Thermal conductivity results for the coatings from three of the papers are presented in Fig. 37.](image)

Figure 37: Thermal conductivity of some of the evaluated TBCs. Data for the sol-gel TBC were supplied by Datec Coating Corp. and the data for Paper E were from Somhorst et al. [135].

5.2.2 Optical properties

The RT hemispherical reflectance values of YSZ TBCs with different types of microstructures and with three different metallic coatings on top of the ceramic are shown in Fig. 38. The reduced reflectance at a wavelength of around 3 µm is due to OH absorption [12, 38, 119]. The OH is incorporated into the coating during spraying, but can disappear during high temperature operation or heat treatment [136]. Except for the OH absorption, there is a general trend towards reduced reflectance at higher wavelengths. This is related to a relative decrease in the size of the defects within the TBCs that cause the scattering, compared with the wavelength of the thermal radiation. It has also been shown by Wang [38] that there is a large increase in the absorptance of plasma-sprayed YSZ at wavelengths above 6 µm, which contributes to the decreasing trend of the reflectance.

The temperature dependence of the reflectance at a selected wavelength, 2 µm, is shown in Fig. 39. It is apparent that the type of microstructure greatly influences the reflectance of YSZ TBCs. TBCs with nanostructured and conventional microstructures had higher reflectance than did those with segmented microstructures. A PS-PVD coating with a columnar microstructure had a relatively poor reflectance at low temperature, but increasing reflectance with increasing temperature, and was the coating with the highest reflectance at temperatures above 650°C. The increase in reflectance with temperature was probably related to oxygen diffusing into the coating and reducing the number of
oxygen vacancies in the ceramic. Fewer oxygen vacancies lead to lower absorbance and consequently higher reflectance [137]. PS-PVD coatings can be expected to have more oxygen vacancies than APS coatings, due to the almost oxygen-free atmosphere in the PS-PVD process. Therefore, a similar high temperature behaviour is not seen for the APS coatings.

The metallic coatings (i.e., Ag, Cr, and FeCrAlY) had relatively low reflectance at the short wavelengths that are most important in a diesel engine. However, the silver coating had a slightly higher reflectance than did the segmented YSZ TBC.

![Figure 38](image_url) Room temperature reflectance of TBCs evaluated in Paper C.

![Figure 39](image_url) Reflectance at 2.0-µm wavelength at elevated temperatures evaluated in Paper C.

5.2.3 In situ heat flux measurements for the combustion chamber

Two types of heat flux measurements were performed. First, heat flux measurements were performed for all the different TBCs to determine the initial heat flux. Then longer running-in tests were performed for three of the coatings, to determine the impact of engine exposure on the heat flux.
5.2.3.1 Initial heat flux

The normalized heat flux with and without TBC on a probe measuring the heat flux through the combustion chamber wall is shown in Fig. 40. YSZ coatings with different types of microstructure (i.e., conventional, nanostructured, and segmented) reduced the flux by 0.2–1.8% versus that of an uncoated steel reference. A top coat consisting of LZ was about as effective as was conventional YSZ in reducing the heat flux. GZ was the most efficient TBC, probably due to a combination of low thermal conductivity and high reflectance, and reduced the heat flux by 4.7%. Sealing the pores of the conventional YSZ coating with aluminium phosphate resulted in higher heat fluxes than that of the steel reference, possibly due to the decreased reflectance of the coating and consequently increased radiative heat transfer through it.

The surface roughness of the various coatings was measured, as this factor can influence the heat transfer (see Table 5). The segmented YSZ coating had the lowest arithmetic average of the roughness profile (Ra value), which may be a factor that explains why that coating had lower heat flux than the other YSZ coatings, despite having higher thermal conductivity and lower reflectance.

The conventional and nanostructured YSZ coatings had the highest Ra values. The high surface roughness of these, and most of the other TBCs, can partly explain why the reduction in heat flux versus that of the steel reference was relatively low.

![Figure 40: Normalized initial heat flux values after correction for varying heat loads (from Paper D).](image)

![Table 5: Surface roughness parameters of coatings in as-sprayed condition (from Paper D).](table)

### Table 5: Surface roughness parameters of coatings in as-sprayed condition (from Paper D).

<table>
<thead>
<tr>
<th>TBC system</th>
<th>Rz [µm]</th>
<th>Ra [µm]</th>
<th>Rk [µm]</th>
<th>Rpk [µm]</th>
<th>Rvk [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional YSZ</td>
<td>65.7</td>
<td>11.2</td>
<td>34.3</td>
<td>21.8</td>
<td>16.6</td>
</tr>
<tr>
<td>Sealed YSZ</td>
<td>34.0</td>
<td>5.5</td>
<td>16.2</td>
<td>4.1</td>
<td>18.2</td>
</tr>
<tr>
<td>Nano YSZ</td>
<td>60.8</td>
<td>10.8</td>
<td>38.4</td>
<td>16.8</td>
<td>15.8</td>
</tr>
<tr>
<td>Segmented YSZ</td>
<td>24.0</td>
<td>4.0</td>
<td>13.7</td>
<td>6.8</td>
<td>5.9</td>
</tr>
<tr>
<td>LZ</td>
<td>38.8</td>
<td>5.6</td>
<td>17.0</td>
<td>9.5</td>
<td>11.5</td>
</tr>
<tr>
<td>GZ</td>
<td>38.5</td>
<td>6.3</td>
<td>22.5</td>
<td>15.8</td>
<td>6.7</td>
</tr>
</tbody>
</table>

5.2.3.2 Running-in of TBCs

The running-in behaviour, in terms of heat flux, of the various top coat materials is shown in Fig. 41. The insulation properties of a top coat consisting of plasma-sprayed YSZ with conventional microstructure improved over time. This was attributed to the growth of microcracks, perpendicular to the direction of the heat flux, near the bond coat (Fig. 36). Also, GZ developed improved thermal insulation properties over time, but this was due to the growth of a large crack in the top coat. Continued exposure to the combustion chamber would soon have resulted in the spallation of much of the top coat. The LZ top coat did not display any beneficial running-in behaviour. The running-in time for the YSZ as well as GZ top coats was about 3 h.
5.2.4 Thermal modelling

The temperature decreases beneath TBC layers of different thicknesses and thermal conductivities inside a straight pipe were calculated according to a method presented elsewhere [13]. The temperature modelling was done for stationary conditions with boundary conditions typical of cast iron manifolds.

A temperature decrease of 50°C in the metallic substrate is required to extend the lifetime of exhaust components [17, 138]. As can be seen in Fig. 42, a top coat thickness of 1.4 mm is needed for a typical plasma-sprayed conventional YSZ TBC with a thermal conductivity of 0.75 Wm\(^{-1}\) K\(^{-1}\) to achieve a 50°C decrease in temperature. For top coat materials with higher thermal conductivity, the coating thickness quickly becomes too great to be practical. For example, mullite, with a thermal conductivity of 2.7 Wm\(^{-1}\) K\(^{-1}\), requires a top coat more than 5 mm thick to obtain a 50°C temperature reduction.

Thermal modelling using the same boundary conditions was also used to estimate to what extent the exhaust test rig, which was used for the thermal cycling of coated pipes (described in section 4.1.2.2), could be used for measuring the effectiveness of different TBCs. It was found that for a typical TBC with a thermal conductivity of 1 Wm\(^{-1}\) K\(^{-1}\) and a thickness of 1 mm, two TBCs with a 10% difference in thermal conductivity could be separated by measuring the outside temperature of the pipes with type-N thermocouples. There are other measurement techniques, such as laser flash, that are better at measuring the thermal conductivity of a material. However, the main benefit of an in situ method is that the combined effect of thermal conductivity, surface roughness, pressure, turbulence, soot deposits, etc., on the insulation effectiveness is measured.
5.3 Engine testing of coated pistons

The results for the engine-tested TBC pistons are presented in this section, divided into temperature measurements and ISFC results.

5.3.1 Temperature measurements

The temperature of the cooling oil, measured when flowing out of the piston cooling galleries, for the different pistons is shown in Fig. 43 for a start of injection (SOI) sweep. The TBCs with the lowest oil temperatures, and consequently the most effective in reducing the heat losses through the piston, were the SPS GZ and SPS YSZ TBCs. The APS YSZ was significantly less effective as a thermal barrier under most of the tested engine conditions.

The temperature in the cylinder head, measured close to the combustion chamber, increased for all three tested TBCs (Fig. 44). The largest increase in temperature, 7.3°C, was seen for APS YSZ.

Also, the exhaust temperature increased for all three tested TBCs (Fig. 45), with the largest increase, 12.6°C, for SPS YSZ. Increased exhaust temperature can be beneficial for engine efficiency if the extra energy can be recovered by the turbine or waste heat recovery systems.

The temperature results indicate that the TBCs are effective in reducing the heat losses through the piston, but result in increased heat losses through the cylinder head and the exhaust. The increased heat losses through the cylinder head and the exhaust can be either due to hotter conditions inside the combustion chamber caused by the TBC, or due to altered combustion. Analysis of the heat release rate (Table 6) shows that the heat release is similar for the TBCs as for the steel piston in the first part of the combustion cycle, but that the last 10% of the fuel is burnt later for the YSZ coatings. The later combustion may be related to various factors, such as altered turbulence due to the rougher surface or fuel trapped within the porous coatings.
Figure 43: Piston cooling oil temperatures for the uncoated and coated pistons for different start of injection (SOI) values (from Paper E).

Figure 44: Temperatures of the cast iron cylinder head close to the combustion chamber, for tests with uncoated and coated pistons (from Paper E).

Figure 45: Exhaust gas temperatures for tests with uncoated and coated pistons (from Paper E).
Table 6: The crank angle after top dead centre (ATDC) where 10, 50, and 90% of the fuel is burnt, for SOI –3°. Standard deviation is only shown for the uncoated steel piston. For the coated pistons, having two measurements per operating point, only the average value is shown (from Paper E).

<table>
<thead>
<tr>
<th>TBC system</th>
<th>10% burn [°ATDC]</th>
<th>50% burn [°ATDC]</th>
<th>90% burn [°ATDC]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Uncoated steel</td>
<td>6.0 ± 0.2</td>
<td>13.1 ± 0.1</td>
<td>30.7 ± 0.6</td>
</tr>
<tr>
<td>APS YSZ</td>
<td>5.9</td>
<td>13.1</td>
<td>34.6</td>
</tr>
<tr>
<td>SPS YSZ</td>
<td>5.9</td>
<td>13.0</td>
<td>33.5</td>
</tr>
<tr>
<td>SPS GZ</td>
<td>5.9</td>
<td>13.0</td>
<td>31.2</td>
</tr>
</tbody>
</table>

5.3.2 ISFC
The indicated specific fuel consumption (ISFC) values for the tests of the different pistons under full load conditions are presented in Fig. 46. The two YSZ TBCs, produced with APS and SPS, both had significant increases in ISFC at all test points except SOI 1°. The largest increase, 2.8%, was measured for SPS YSZ at SOI –5°. SPS GZ did not display any significant increase in ISFC.

Figure 46: Indicated specific fuel consumption under full load conditions for tests of uncoated and coated pistons. GZ cannot be separated from the steel reference due to the large spread in the results (from Paper E).

5.4 Summary of TBC effectiveness
A summary of the effectiveness of the different TBCs as thermal barriers is presented in Table 7. The table contains the results of the measurements of thermal conductivity and optical properties, heat flux probe results, temperature measurements, and ISFC results of the piston tests. The colour markings in the table show how the different evaluation methods and results are connected to RQs 4–8. The connections between the results and the RQs are further discussed in chapter 6.
Table 7: Summary of the TBC effectiveness results in comparison with the reference, APS conventional YSZ. Grading: 0 similar to reference, ++ much better, + better, - worse, - - much worse response than reference. Colour markings show the connections to RQ4, RQ5, RQ6, RQ7, and RQ8.

<table>
<thead>
<tr>
<th>Top coat material</th>
<th>Coating process</th>
<th>Top coat microstructure (+ surface modification)</th>
<th>Effectiveness as thermal barrier</th>
</tr>
</thead>
<tbody>
<tr>
<td>YSZ</td>
<td>APS</td>
<td>Conventional</td>
<td>0</td>
</tr>
<tr>
<td>APS</td>
<td>Conv. (+ Ag)</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>APS</td>
<td>Conv. (+ Cr)</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>APS</td>
<td>Conv. (+ FeCrAlY)</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>APS</td>
<td>Conv. (+ sealing)</td>
<td>0</td>
<td>+</td>
</tr>
<tr>
<td>APS</td>
<td>Nanostructured</td>
<td>0</td>
<td>+</td>
</tr>
<tr>
<td>APS</td>
<td>Segmented</td>
<td>0</td>
<td>-</td>
</tr>
<tr>
<td>PS-PVD</td>
<td>Columnar</td>
<td>0</td>
<td>+</td>
</tr>
<tr>
<td>SPS</td>
<td>Columnar</td>
<td>0</td>
<td>+</td>
</tr>
<tr>
<td>CaSZ/YSZ</td>
<td>Sol-gel (sprayed)</td>
<td>0</td>
<td>+</td>
</tr>
<tr>
<td></td>
<td>Sol-gel (dipped)</td>
<td>0</td>
<td>+</td>
</tr>
<tr>
<td>GZ</td>
<td>APS</td>
<td>Conventional</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td>SPS</td>
<td>Columnar</td>
<td>0</td>
</tr>
<tr>
<td>LZ</td>
<td>APS</td>
<td>Conventional</td>
<td>0</td>
</tr>
</tbody>
</table>

*) Suspected crack from sample preparation
**) Evaluated at temperatures below 600°C
6 Discussion, conclusions, and future work

In the discussion part of this chapter, the results and answers to the RQs are first discussed. This is followed by a discussion from a systems perspective: first treating what TBCs mean to the diesel engine as a system, and then treating what impact TBCs in diesel engines may have from a global perspective based on the UN Global Goals for sustainable development. The novelty of the research and how the results can be used in the industry are also discussed. Finally, conclusions are drawn from the research and suggestions are made for future work.

6.1 Discussion

RQ1: How do different coating microstructures, produced using APS and PS-PVD, influence the thermal cycling lifetime of YSZ TBCs in low cycle fatigue tests?

The results of the different TCF tests (Table 4) indicate that the type of microstructure greatly affects the lifetime of YSZ TBCs when these are exposed to conditions typical of heavy-duty diesel engines.

APS coatings with conventional microstructure outperform the conventional and nanostructured YSZ APS coatings by a factor of 5–10 in the cyclic furnace test with forced cooling (Fig. 31). The columnar microstructure of the PS-PVD coating survived the same TCF test even longer, but the better oxidation resistance of the bond coat and lower thickness of the top coat made it difficult to make a fair comparison with the APS coatings. However, it appears as though the high strain tolerance caused by the cracks perpendicular to the substrate in the segmented APS coating, and by the gaps between the columnar grains in the PS-PVD coating, has a positive effect on the thermal cycling lifetime. This is in agreement with previous work using other types of TCF testing, in which long thermal cycling lifetimes have been observed for APS coatings with segmentation cracks [62, 69] and for PS-PVD coatings with intercolumnar gaps [71, 72].

The thermal cycling lifetime of plasma-sprayed nanostructured YSZ was compared with that of plasma-sprayed conventional YSZ in four different low cycle fatigue tests. The nanostructured YSZ performed slightly better than did conventional APS coatings in two out of four tests, and gave similar results in the remaining two tests. One factor contributing to difficulties to differentiate between the lifetimes of the two coatings in one of these tests was the severe oxidation of the FeCrAlY bond coat. To resolve this, shorter inspection intervals could have been used. However, these results indicate that nanostructured plasma-sprayed YSZ may have a slightly better thermal cycling lifetime than plasma-sprayed YSZ with conventional microstructure when used for exhaust manifolds in heavy-duty diesel engines. These findings are similar to what others have found in TCF tests at higher temperatures and with superalloys instead of cast iron (or steel) as substrate materials [62, 63]. It has been shown that some nanostructured TBCs have a lower elastic modulus than do conventional TBCs, which leads to reduced stresses [62, 63]. This is possibly the reason for the better performance of the nanostructured YSZ TBC seen in some of the TCF tests in this work.

The severe oxidation (Fig. 33) of the FeCrAlY bond coat in the thermal cycling furnace test, with 800°C as the maximum temperature, indicates that a bond coat with higher oxidation resistance is required at such temperatures. The bond coat may reach temperatures exceeding 700°C for TBCs applied to the inner surface of exhaust and turbo manifolds. Bond coats in which iron has been replaced with nickel or nickel/cobalt would be better for these components due to their improved high-temperature oxidation resistance. However, oxidation rates of FeCrAlY have been found to be rather low at temperatures up to 600°C [22]. The TBCs evaluated in the exhaust test rig, where they were exposed to exhaust gas temperatures of 530°C, had no TGO on the bond coat. For combustion chamber components with active cooling and consequently relatively low bond coat temperatures, such as the piston and cylinder head, FeCrAlY is expected to perform well as the bond coat.

The different types of microstructure are likely to spall in different ways once they fail. The PS-PVD coating with its columnar structure, with weak or no bonding between the individual columnar grains, is likely to fracture into smaller fragments before reaching the turbocharger. The associated
risk of damage to the turbine wheel blades is therefore considered to be lower for the columnar than the other investigated types of microstructure.

\textbf{RQ2: What is the thermal cycling performance of different TBCs like for exhaust manifold applications?}

As was discussed for RQ1, the type of microstructure has a large influence on the TCF life of YSZ TBCs. It is also of interest to study the thermal cycling lifetime of other top coat materials than YSZ and other coating processes than APS.

YSZ performed the best in the TCF tests of the tested top coat materials with conventional APS microstructure, while LZ and forsterite performed slightly worse. The worst performing top coat material was mullite. Two of these top coat materials, mullite and LZ, have a lower thermal expansion than that of YSZ and consequently a larger thermal expansion mismatch with the substrate material. This will cause higher stresses and strains during thermal cycling, probably contributing to the poor performance. Both forsterite and mullite had relatively low porosity and an almost total absence of interlamellar cracks. Interlamellar cracks and pores reduce the elastic modulus of the coatings and improve their strain tolerance \[10, 14\]. The thermal cycling lifetimes of mullite and forsterite TBCs could probably be improved by increasing the porosity and number of interlamellar cracks.

The two sol-gel composite coatings, which incorporated CaSZ/YSZ particles, had a good thermal cycling lifetime, attributable to vertical cracks that improved the strain tolerance of the coatings. The lifetime of these coatings, as well as the possibility of using this coating process for the internal coating of narrow pipes, makes them promising candidates for exhaust manifolds. However, further development of these TBCs is required in order to reduce their thermal conductivity before they can be a viable alternative for such applications.

Oxidation of the cast iron substrate was found in all APS TBCs after the TCF tests, independent of the bond coat and top coat material used. The relatively high porosity, due to the APS process used for depositing the bond coat, likely contributed to the low oxidation protection. The only cast iron substrate that was not oxidized during the TCF tests was that beneath the sol-gel top coats, likely due to lower oxygen diffusion through the denser top coat. The oxidation resistance could also be improved by making the bond coat denser, for example, by using the high-velocity oxygen fuel process.

It is important to remember that there are limitations in how far into narrow pipes TBCs can be applied by thermal spraying. For some components it may be sufficient to coat only the end sections of the manifolds, where it is possible to use for example APS. To be able to apply a TBC further into a manifold, it might be required to either use another coating method, such as slurry or sol-gel, or designing the manifold into two parts. If the manifold is divided into two parts, these could be coated by e.g. APS before being mounted together.

\textbf{RQ3: How are different TBCs influenced by the thermal cycling fatigue exposure inside the combustion chamber?}

The TBCs evaluated inside the combustion chamber were mainly exposed to high cycle fatigue, as these were tested under a large number of combustion events but with only a few starts/stops. The fast cycling in combination with oil/water/compressed air cooling of the substrate material during the test gives temperature fluctuations near the top coat surface and rather stable temperatures near the bond coat. Despite the relatively small number of cycles for a high cycle fatigue test, some initial signs of failure were observed.

No TBC performed better than did the APS YSZ with conventional microstructure, in either the piston test or the running-in heat flux test. The piston test showed no influence at all on this TBC, while the running-in test showed the growth of some microcracks within the top coat (Fig. 36c).

SPS YSZ also seems promising from a lifetime perspective. The SPS YSZ piston had only a few small pits in the surface (Fig. 36a and b). These could be related to problems obtaining a uniform coating
quality for the complicated geometry, rather than to the engine exposure. The columnar type of microstructure of the SPS YSZ coating displayed no other problems during the piston test, displaying good high cycle fatigue performance for this type of microstructure inside the combustion chamber of a diesel engine.

GZ produced with APS as well as SPS displayed some lifetime issues in both of the high cycle fatigue tests. In the running-in test, large cracks formed within the APS GZ top coat close to the top coat/bond coat interface (Fig. 36d). The SPS GZ piston had some lateral cracks within the top coat, which may have been formed either during the coating process or during the engine exposure. In TCF tests, GZ has been shown by others to be less tough and have a shorter lifetime than YSZ [27]. In TCF tests performed at much higher temperatures, double-layer systems, with a first layer of YSZ providing toughness and a second layer of GZ providing low thermal conductivity, have been shown to improve the lifetime compared with a single GZ layer as top coat [27]. Double-layer YSZ/GZ systems can also be expected to reduce the risk of fractures when exposed to high cycle fatigue inside the combustion chamber of a diesel engine.

RQ4: How is the substrate temperature of exhaust components influenced by different TBCs and different thicknesses of the coating?

A lower cast iron temperature would prolong the lifetime of the exhaust and turbo manifolds, due to a lower oxidation rate as well as reduced risk of problems with thermo–mechanical fatigue. A targeted 50°C decrease in the temperature of the metal beneath the TBC has been called for in earlier studies [17, 138].

Fig. 42 shows the TBC thickness required in order to achieve different substrate temperature reductions. For example, a plasma-sprayed TBC with a conventional type of microstructure and YSZ or LZ as the top coat material typically has a thermal conductivity of about 0.75 W m⁻¹ K⁻¹ and would need to be about 1.4 mm thick to obtain a 50°C temperature reduction of the substrate beneath the top coat. Such thick coatings may cause durability problems, as these coatings will typically have large residual stresses from the coating process and an increased risk of premature failure [139].

A TBC 0.4 mm thick, for example, can be expected to have a better lifetime than a thicker TBC, and some APS TBCs of that thickness were shown to survive TCF tests without any significant damage. For a 0.4-mm coating thickness, a thermal conductivity of 0.22 W m⁻¹ K⁻¹ would be required to achieve a 50°C reduction (Fig. 42). Such a low thermal conductivity may be difficult to obtain with thermal spraying, but it has been shown that some novel slurry coatings have thermal conductivities close to that value [24]. For a typical APS TBC with a thermal conductivity of 0.75 W m⁻¹ K⁻¹, a temperature reduction of 16°C can be achieved with a 0.4-mm TBC, which is lower than the target set in the earlier studies. However, as shown by finite element calculations for manifolds [13], even such temperature reductions can have a significant impact on the thermo–mechanical fatigue life of a component.

As atmospheric plasma spraying is a line-of-sight process, it cannot be used to coat the entire inner surface of a long narrow pipe. However, by spraying from the ends of a pipe, a TBC can be applied a few centimetres into the pipe. Because some exhaust components have the highest thermal loads close to their ends, coating even these small areas of the components may be enough for a significant improvement in fatigue lifetime. If a TBC is required farther inside the pipe, a coating process such as a slurry or sol–gel process would be more suitable.

It is worth noting that Eqs. 11 and 12, which are used for the thermal modelling, do not take into account how factors such as the increased surface roughness or porosity of the coated surface influence the heat transfer from the gas to the wall. In situ measurements would be required to more accurately determine the effectiveness of different coatings as thermal barriers for manifolds. The method presented in section 4.1.2.2, using thermocouples to measure the outer surface temperature of coated pipes within the exhaust test rig, could be used to measure the effectiveness of the TBCs. An in situ measurement would not rely on the thermal conductivity results of laser flash measurements as input for thermal calculations. Such measurements may have large spreads, as seen in Fig. 37, which may be attributed to measurement errors as well as variations in the microstructure of the coatings produced on different occasions and with different spray guns.
RQ5: How are thermal insulation properties of TBCs influenced by different coating microstructures, produced with APS, SPS, and PS-PVD, and by the addition of thin surface layers?

The nanostructured YSZ APS coating has the highest reflectance of all tested coatings up to 650°C. Temperatures below 650°C have usually been reported from measurements and calculations of the surface temperatures of TBCs inside the combustion chambers of diesel engines [89, 94, 140]. Due to its high reflectance and low thermal conductivity, nanostructured YSZ can be expected to perform better than the other evaluated TBCs inside the combustion chambers of engines, where the influence of thermal radiation is significant. PS-PVD is the coating with the highest reflectance at temperatures above 650°C. Together with relatively low thermal conductivity, this coating has the required properties for pistons, which may reach such temperatures. However, high cost and the high substrate temperature required during the coating process may limit the use of this coating process for diesel engine components. SPS coatings, which have been shown to have promising high cycle fatigue properties inside the combustion chamber, may be a better alternative to PS-PVD for diesel engine applications. SPS coatings also have high reflectance and low thermal conductivity [79].

The reflectance of a conventional YSZ coating with a silver layer on top of the ceramic was relatively low. Of the ceramic coatings, only the segmented YSZ coating had lower reflectance than that of the silver-coated TBC. By polishing the YSZ coating to a smoother surface before applying the silver layer, and by ensuring that the silver deposition process produces a smooth surface, the reflectance could be enhanced still further. The silver coating was not tested in an engine and there is a risk that its reflectance will not remain stable over time in that environment. YSZ with chromium or FeCrAlY surface layers had the lowest reflectance of all evaluated TBCs.

The conventional and nanostructured APS TBCs had the lowest thermal conductivities – less than half that of the segmented APS coating and slightly lower than the literature data on PS-PVD coatings [72, 141]. Conventional and nanostructured APS coatings therefore have the types of microstructure that, compared to the other investigated TBCs, provide the best thermal insulation inside exhaust components and combustion chambers where the thermal radiation is of only minor importance.

RQ6: How is the heat flux from the combustion chamber influenced by TBCs with respect to their composition, microstructure, and sealed porosity?

The heat flux probe measures the combined effect of thermal transport by conduction and radiation through the TBC. As compared with an uncoated steel reference sample, the heat flux was reduced by 4.7% with a GZ coating, while a conventional YSZ coating and an LZ coating produced almost no decrease in heat flux (Fig. 40). Of these coating materials, dense GZ and LZ both have about 25% lower thermal conductivities than does dense YSZ [34]. Perhaps more relevant are thermal conductivity results for actual TBCs with a porous microstructure. SPS coatings have also been shown to have lower thermal conductivity for GZ than for YSZ [135]. For the APS coatings evaluated in Paper A it was shown that LZ had similar thermal conductivity as YSZ. The ranking between the different TBCs could then to a large extent be explained by the differences in thermal conductivity for sprayed coatings. However, the relatively small difference in heat flux compared to the uncoated steel reference suggest that other factors, such as optical properties or hot gas infiltration into the pores, are of importance. The lower transmittance and higher reflectance of GZ compared to YSZ at wavelengths below 2.7 µm [119] is a factor, which, together with its low thermal conductivity, that could explain why only this TBC shows a reduction in heat flux compared to the steel reference. The slightly greater thickness of the GZ coating, i.e., 410 µm versus 350 and 360 µm for YSZ and LZ coatings, respectively, is another important factor that contributes to the heat flux reduction.

The three YSZ coatings with different microstructures all produced rather small reductions in heat flux from the combustion chamber (Fig. 40). The heat flux reduction was 1.8% for the segmented coating, while the nanostructured and conventional coatings did not significantly reduce the heat flux. The fact that the segmented coating produced the largest heat flux reduction, despite having the highest thermal conductivity and lowest reflectance of the three, indicates that additional factors
influence the heat transfer through the coatings. One factor may be the very low porosity of the segmented coating, which means that a smaller amount of hot gas can infiltrate the coating and heat it at some depth below the surface. The segmented coating also has the lowest surface roughness; this contributes to lower heat losses, as others have demonstrated for similar surfaces [107–111, 142]. The very small extent of the nanozones – only about 0.2 area% – in the nanostructured YSZ coating explains why the heat flux result is about the same as for the conventional YSZ coating. Nanostructured coatings with more nanozones can be expected to be better at reducing the heat flux, as a decreased pore size reduces the radiative heat transfer [88]. The extent of nanozones was in the range 8–16 area% in the other papers (Table 2), and those coatings can therefore be more accurately considered as nanostructured than the one in the heat flux test.

The conventional YSZ coating with the pores sealed with aluminium phosphate produced a significant increase in heat flux (Fig. 40), even though the thermal conductivity of the sealed coating can be expected to be much lower than that of steel. Increased radiative heat transport, because of the sealed pores, is believed to be the one reason for this high heat flux. Infiltrating pores in YSZ coatings with epoxy (refractive index \( n = 1.66 \)) or calcium-magnesium-alumino-silicates (\( n = 1.62 \)) has been demonstrated to reduce the scattering coefficient, and consequently also the reflectance, with a large increase in thermal radiation transmitted though the coating as a result [143, 144]. Filling the pores with aluminium phosphate (\( n = 1.51 \) [145]) may have a similar effect. When the pores are filled with a substance with a higher refractive index than that of air (\( n = 1.00 \)), the scattering will decrease. When the reflectance of the coating decreases, it is likely that more thermal radiation will reach the bond coat and cause deep heating. Sealing the pores may therefore cause more problems than it solves. Having a sealing substance with a low refractive index might reduce the problems of low scattering.

**RQ7: Is there a running-in period, with respect to thermal insulation properties, for TBCs inside the combustion chamber?**

High-temperature exposure may result in changes in thermal conductivity over time due to sintering or the growth of microcracks [92, 93]. The exposure inside the combustion chamber also results in soot and ash deposits on the surface, which influence optical properties and may act as an additional insulating layer [146]. The effect of these changes to the surface and top coat decreases over time until a more steady-state thermal conductivity value is reached. Plasma-sprayed GZ coating as well as plasma-sprayed conventional YSZ coating both had a running-in period of about 2–3 h before a more stable heat flux value was reached (Fig. 41). For the conventional YSZ coating, the heat flux through the TBC decreased over time. Microstructural analysis indicated that interlamellar cracks near the bond coat had increased in length (Fig. 36c). Because these cracks are oriented perpendicular to the heat flux, they are effective in improving the thermal insulation. However, there is a risk that these cracks will continue to grow in size and not only have a positive effect on thermal insulation but also have a negative effect on the lifetime of the coating. For the GZ TBC there was also a reduction in heat flux, but this was due to the growth of a large lateral crack near the top coat/bond coat interface (Fig. 36d). The orientation of this crack was also effective in reducing the heat flux, similar to the interlamellar cracks in the YSZ coating. However, the size of the crack suggests that the coating was close to spalling.

Unlike the other two TBCs, the plasma-sprayed LZ coating did not cause any change in heat flux over time (Fig. 41). A large crack, oriented mainly in the lateral direction, was found in the LZ coating after the 5-h test. It is reasonable to believe that a crack of that size and orientation would result in a decrease in heat flux similar to that produced by the crack in the GZ coating if it had formed during the test. Since there was no decrease in heat flux during the test, the crack probably either existed in the coating before the test or, more likely, formed after the test.

**RQ8: How are the efficiency and heat losses of an engine influenced by different types of TBCs produced with APS and SPS?**

YSZ TBCs produced with APS and SPS both increased the ISFC (i.e., reduced the efficiency) of the engine when applied to the pistons (Fig. 46). An SPS GZ coating, on the other hand, caused no significant increase in ISFC. A small shift in the heat release towards a later part of the cycle could be seen for the YSZ TBCs (Table 6). Similar behaviour has been reported by several others for
insulated combustion chambers and could be related to slower flame propagation due to rougher surfaces and hotter gases [94, 103]. Considering that the results presented in this work are the first regarding SPS coatings on pistons in a heavy-duty diesel engine, and that factors such as surface roughness and sealing layers have not yet been evaluated for these TBCs, the SPS GZ coating would seem to be a promising candidate for future tests.

All three TBCs reduced the piston cooling oil temperature, increased the temperature of the cylinder head, and increased exhaust temperatures (Figs. 43–45) when compared with a standard steel piston without any TBC.

The heat losses for the three different TBCs tested on pistons were qualitatively assessed, with the APS YSZ coating as a baseline (Table 7). This revealed that the SPS YSZ is better than APS YSZ in reducing the piston cooling oil as well as cylinder head temperatures, though it results in higher exhaust temperatures than does the APS YSZ. The ISFC is similar for these two TBCs. The benefits of reduced heat transfer through the SPS YSZ-coated piston and to the head, compared with APS YSZ, are thus counteracted by increased heat loss to the exhaust. Comparing SPS GZ and APS YSZ coatings shows that the piston cooling temperatures and cylinder head temperatures are lower for SPS GZ, while the exhaust temperatures are almost the same. In this case, the reduced heat losses inside the combustion chamber are not counteracted by increased exhaust temperatures, and SPS GZ produces a lower ISFC than does APS YSZ.

These eight RQs can be summarized in light of the main goal of the thesis:

_**How can thermal barrier coatings be designed for improved lifetime and insulation effectiveness for combustion chamber and exhaust components in a heavy-duty diesel engine?**_

The results of the different experiments indicate that the TBCs with the longest thermal cycling lifetimes usually do not have the best insulation properties and vice versa. GZ has promising thermal properties, but seems to have a limited lifetime in heavy-duty diesel engines, as has been shown in TCF tests at higher temperatures [27]. TBCs with a columnar microstructure or segmentation cracks are the most promising from a thermal cycling lifetime perspective, but may have limitations when it comes to thermal properties. Since the requirements regarding insulation effectiveness as well as TCF exposure differ slightly between manifolds and combustion chamber components, these areas are discussed separately below.

**Manifold insulation effectiveness**

The TBC inside a manifold needs to have a rather thick top coat to significantly reduce the substrate temperature. A compromise needs to be made between lifetime and insulation requirements for a specific engine, before choosing what type of microstructure to use. Nanostructured APS YSZ has lower thermal conductivity than does, for example, segmented APS YSZ. SPS and slurry types of TBCs have not been evaluated within this thesis for manifold applications, but have some interesting properties and would be of interest for future work.

**Manifold lifetime**

The results indicate that the bond coat oxidation is low for exhaust components in heavy-duty diesel engines, but that the cast iron substrate is sensitive to oxidation due to oxygen transport through the TBC. When designing a TBC to be used inside exhaust components, it is important to ensure low oxygen transport through the bond coat, for example, by changing the chemical composition of the bond coat or reducing its porosity. Considering the lifetime of TBCs for manifolds, segmented or columnar types of microstructures perform the best and YSZ was the best performing top coat material.
Combustion chamber insulation effectiveness

The TBC material with the best insulation effectiveness, in both the heat flux probe test and the piston test, was GZ. The good performance of this material can be explained by a combination of high reflectance and low thermal conductivity. It was shown in the piston test that SPS YSZ insulated the piston better than did APS YSZ, even though it had higher thermal conductivity. The type of microstructure seems to be important for thermal barrier performance for a component where the flame hits the surface.

The heat flux probe testing, on the other hand, showed that the type of microstructure did not have any large influence on the insulation effectiveness. The type of microstructure seems to be less important for a surface that is not in contact with the flame.

Combustion chamber lifetime

The TBC exposed to high cycle fatigue inside the combustion chamber displayed varying degrees of damage. YSZ produced better results than did GZ or LZ, as reported by others for tests at higher temperatures [27]. In the heat flux test, conventional YSZ displayed some growth of microcracks due to the engine exposure (Fig. 36c), while SPS YSZ on pistons had some pits (Fig. 36a and b), which could be related to problems during the spraying process. If the spraying process can be optimized, SPS YSZ seems as the most promising TBC of those tested, considering high cycle fatigue lifetime.

Considering the promising results of GZ in terms of insulation effectiveness inside the combustion chamber, optimizing TBCs with this type of top coat material would be of considerable interest. Double-layer systems with YSZ as a first layer providing toughness and GZ as a second layer providing improved thermal properties should be evaluated in future tests. Such double-layer TBCs have been shown to have significantly improved thermal cycling lifetimes [27].

System perspective – engine

It is always important to consider the engine as a system if applying a TBC to some components. For example, the tests of different TBCs on the pistons showed that the temperature of the cylinder head increased (Fig. 44). This may lead to TCF problems and a need, for example, to apply a TBC to that component as well, or to improve the cooling of the head. An increased exhaust temperature was also seen in the test with coated pistons (Fig. 45), which can have positive as well as negative effects on the engine. It is positive in that there is more energy in the exhaust to be recovered by the turbocharger or waste heat recovery systems. The higher temperature of the manifolds is a negative effect, because even a small difference in temperature can have a relatively large influence on the thermo-mechanical fatigue life of the component [13], unless also the manifold is internally coated with TBC.

Another consequence of TBCs inside the combustion chamber and the hotter conditions there is the potential deterioration of lubrication oil properties [115]. This may lead to an increase in wear and friction in the engine, for example, in the piston ring/cylinder liner contact.

Reduced heat losses to the coolant or to the oil in an insulated engine also means that there is a potential to reduce the size of the cooling system. That would lead to lower weight, which for a heavy-duty truck means that it can carry a larger load, as well as reduced costs and parasitic losses for the engine.
**Global perspective: UN Global Goals**

The UN has set 17 global goals for the sustainable development of our civilization. This thesis is mainly related to the goals concerning *climate action* and *responsible consumption and production*.

**Climate action**

Previous research has had varying degrees of success in determining how much engine efficiency can be improved for insulated engines. Some early research into TBCs found improvements of up to 6% for heavy-duty diesel engines [9], while more recent research has found smaller improvements or even reduced efficiency. Reduced efficiency was also seen for some of the TBCs examined here, although the reduction was relatively small for the novel type of SPS GZ TBC. It is believed that, with the renewed interest in TBC research in many organizations around the world, a reduction of fuel consumption by 1–2% could be achieved in heavy-duty diesel engines. Even small reductions in fuel consumption will lead to large reductions in CO₂ emissions, considering that global CO₂ emissions from heavy-duty trucks and buses were 1400 million tonnes in 2012 [147].

**Responsible consumption and production**

The use of TBCs can be related to the responsible consumption and production goal as they enable the improved lifetime of engine components and lower fuel consumption. The reduced heat flux into insulated components means reduced problems with oxidation and thermo–mechanical fatigue, which are factors that may limit these components’ fatigue life. Improved lifetime can be expected for many components, reducing the number of spare parts and the resources required during the engine lifetime. As discussed above, it is important to consider the entire engine as a system, as one coated component might cause increased temperatures and reduced lifetime for another component.

When designing a TBC, it is important to consider how the material can be recycled once the engine is taken out of service. There is a risk that some TBC materials will contaminate the steel when the coated components are recycled and melted. Non-oxidized aluminium, present in bond coats, may degrade the properties of the recycled steel. There is less risk with, for example, zirconia- or alumina-based top coats, as these will float to the surface of the melt, due to their low densities, where they can more easily be separated. Any TBC system should be evaluated also from this perspective.

**How can the results be used?**

The results presented here can help in designing TBCs for diesel engines. Some of the novel test methods presented here can also be of great value when evaluating new types of TBCs.

- **Thermal cycling results** indicate how different types of microstructures and top coat materials behave under thermal cycling conditions typical of heavy-duty diesel engines. This is of value when selecting what TBC to use for different engine components in the combustion chamber of the exhaust system. The present summary of the thermal cycling lifetimes of different TBCs is much more extensive than has previously been presented in the literature on heavy-duty diesel engine applications.

- Similarly, the results concerning the effectiveness of different types of TBCs as thermal barriers in heavy-duty diesel engines can help in designing TBCs for the conditions inside the engine. The conditions within a diesel engine are quite complex, including rapid thermal cycling, soot, and large variations over the cycle in thermal radiation, pressure, and gas flow. These factors make it difficult to predict TBC effectiveness based solely on lab tests, so lab results need to be complemented by in situ test results, as in this work.

- The in situ heat flux test method developed here for evaluating the effectiveness of TBCs inside the combustion chamber indicated that GZ performs better than YSZ, which is also what is seen in the engine test with coated pistons. The in situ heat flux test method thus
seems promising for the relatively simple evaluation of coatings before testing them on real components. The lab tests of thermal conductivity and optical properties are not considered to provide sufficient data to predict the performance inside the combustion chamber.

- The in situ heat flux test method was used to examine the running-in behaviour of some coatings. It has been shown that, for the specific engine conditions used here, the heat flux was reduced for the first 2–3 h of engine exposure. These results show the importance of being aware of the running-in period and running-in behaviour of the TBCs when evaluating different coatings in engine tests, to avoid incorrect results and conclusions from engine tests.

- The in situ test method presented here for evaluating the thermal cycling lifetimes of TBCs inside manifolds can be of great value when testing new TBCs for exhaust components. This test method can be used in testing the thermal cycling lifetimes of several TBCs simultaneously, under realistic conditions and with realistic geometries, without any risk of damaging the engine if some coatings spall off. The possibility of evaluating many coatings in a single engine test means that it is a very time- and cost-effective method compared with full-scale engine tests, in which TBCs are applied to real components. Thermal cycling testing in furnaces seems to give results similar to those of in situ testing, and could therefore be an even easier method for evaluating the thermal cycling lifetimes of TBCs; however, one advantage of the in situ method is that it can be used with thermocouples to evaluate different coatings’ effectiveness as thermal barriers.

- Tests of SPS TBCs in heavy-duty diesel engines have not previously been reported, and the results of such tests conducted here are promising.

6.2 Conclusions

*RQ1: How do different coating microstructures, produced using APS and PS-PVD, influence the thermal cycling lifetime of YSZ TBCs in low cycle fatigue tests?*

The type of microstructure greatly affects the lifetime of YSZ TBCs when these are exposed to conditions typical of heavy-duty diesel engines. The segmented APS coating displayed the best performance of the APS coatings, only outperformed by the PS-PVD coating.

*RQ2: What is the thermal cycling performance of different TBCs like for exhaust manifold applications?*

YSZ performed the best in the TCF tests of the tested top coat materials with conventional APS microstructure. LZ and forsterite performed slightly worse, followed by mullite. The two sol-gel composite coatings, which incorporated CaSZ/YSZ particles, had good thermal cycling lifetimes.

*RQ3: How are different TBCs influenced by the thermal cycling fatigue exposure inside the combustion chamber?*

APS YSZ displayed the best high cycle fatigue resistance when exposed inside the combustion chamber. The only evident influence on the coatings was the growth of some interlamellar cracks after a running-in test. SPS YSZ also seems promising from a lifetime perspective. GZ produced with APS as well as with SPS displayed some lifetime issues in the high cycle fatigue tests.

*RQ4: How is the substrate temperature of exhaust components influenced by different TBCs and different thicknesses of the coating?*

A plasma-sprayed TBC with a conventional type of microstructure and YSZ or LZ as the top coat material with a typical thermal conductivity of 0.75 W m\(^{-1}\) K\(^{-1}\) would require a thickness of about 1.4 mm to obtain a 50°C temperature reduction of the substrate beneath the top coat. For \(k = 0.75\) W m\(^{-1}\) K\(^{-1}\), a temperature reduction of 16°C can be achieved with a 0.4-mm TBC.
RQ5: How are thermal insulation properties of TBCs influenced by different coating microstructures produced with APS, SPS, and PS-PVD, and by the addition of thin surface layers?

The TBC with the best thermal insulation properties for the combustion chamber was the nanostructured APS coating, followed by the conventional APS coating, since these had a combination of low thermal conductivity and high hemispherical reflectance. Reflectance was not improved by adding a thin metallic surface layer. The TBCs expected to perform the best inside manifolds are conventional and nanostructured APS TBCs due to their low thermal conductivities.

RQ6: How is the heat flux from the combustion chamber influenced by TBCs with respect to their composition, microstructure, and sealed porosity?

In situ heat flux measurements of plasma-sprayed TBCs showed that GZ was the coating material that provided the best thermal insulation, while YSZ with different types of microstructure and LZ were less effective. The influence of microstructure type was found to be small. Plasma-sprayed YSZ coatings with the pores sealed with aluminium phosphate had higher heat fluxes than that of a steel reference.

RQ7: Is there a running-in period, with respect to thermal insulation properties, for TBCs inside the combustion chamber?

There is a running-in period for plasma-sprayed YSZ and GZ coatings. The heat flux through these two TBCs decreased during the first 2–3 h before stabilizing.

RQ8: How are the efficiency and heat losses of an engine influenced by different types of TBCs produced with APS and SPS?

YSZ TBCs produced with APS and SPS both increased the ISFC of the engine when applied to the pistons. SPS GZ, on the other hand, resulted in no significant increase in ISFC. All three TBCs reduced the temperature of the piston cooling oil, increased the temperature of the cylinder head, and increased the exhaust temperatures compared with those of a standard steel piston without any TBC.

By summarizing these RQs in light of the main research question of the thesis, “How can thermal barrier coatings be designed for improved lifetime and insulation effectiveness for combustion chamber and exhaust components in a heavy-duty diesel engine?”, the following conclusions can be drawn:

The TBCs inside manifolds need to have a rather thick top coat to be able to provide a significant reduction in substrate temperature. A compromise needs to be made between lifetime and insulation requirements for a specific engine, before choosing what type of microstructure to use. Nanostructured APS YSZ has lower thermal conductivity than does, for example, segmented APS YSZ, but also a shorter lifetime. Segmented or columnar types of microstructures have the longest thermal cycling lifetimes. When designing a TBC to be used inside exhaust components, it is also important to ensure low oxygen transport through the bond coat to protect the substrate from oxidation.

The TBC material with the best insulation effectiveness inside the combustion chamber was GZ, but it has a shorter thermal cycling lifetime than does YSZ. It was shown that SPS YSZ insulated the piston better than did APS YSZ, even though it had higher thermal conductivity. The type of microstructure seems to be important for the thermal barrier performance of a component where the flame hits the surface, while the heat flux probe measurements indicated almost no influence of microstructure on TBC effectiveness for a surface that is not in contact with the flame.
6.3 Future work

In the engine test with coated pistons, the SPS TBCs displayed promising performance, so further development of this type of TBC would be of interest. Parameters that can be improved through future work include smoother surfaces, sealing layers, and double-layer systems with, for example, a first layer of YSZ and a second layer of GZ.

The heat flux probe measurements indicated increased heat flux when adding a sealing layer to the TBC, and the mechanism behind this needs to be further investigated. The optical properties of the sealed surface could be one explanation. However, the interaction between the flame and the TBC surface is relatively unexplored, so more research is required. Experiments with different types of sealing layers or surface coatings, including measurements of optical properties of these coatings, could reveal more about how to optimize the design of the TBC surface.

The tests of TBCs for manifolds revealed problems with oxidation of the substrate material. Since this may be a limiting factor for the lifetime of the TBC, it is important to improve the oxidation resistance of the bond coat by making it denser or changing its chemical composition.

SPS and slurry coatings have not been evaluated for manifolds within this thesis, but they have some interesting advantages compared with APS coatings. SPS can produce columnar coatings with a combination of low thermal conductivity and long thermal cycling lifetime, while it is easier to use slurry coating for the internal surfaces of manifolds.
Reference list


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