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Fakulta strojní

Ústav letadlové techniky



Thermal Spray Coatings and their mechanical testing

Bakalářská práce

Studijní program: Strojírenství

Studijní obor: Konstruování podporované počítačem

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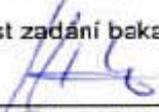
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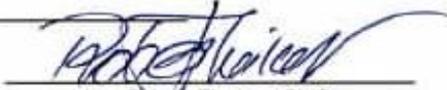
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Abstract

Thermal spray coatings are rapidly growing spraying technique in the fields of manufacturing, e.g. aerospace, chemical and petrochemical industry, automotive, medicine and biomedicine. Among others, thermal spray coatings include thermal barrier coatings which are refractory layers protecting the base material of parts operating at extreme high temperatures. Typical products using thermal barrier coatings are turbine vanes, machinery parts but also prostheses. TBCs provide protection from heat, wear and corrosion, which is why are they used worldwide for a variety of applications.

This writing is intended to provide general information about thermal barrier coatings and gradually resolve several issues. At the beginning of writing, I will provide information about types of processes and principles used for thermal barrier coatings and their performance. Then we will move on to the part in research in which we will endeavour to find out the best option for use of thermal barrier coatings in aerospace, concretely in an inner casing of a flame tube in a combustion chamber of an aircraft engine. Due to cooperation, GE Aviation Czech will provide samples used in the combustion chamber of the aircraft engine, which we will need for testing. In the experimental part, samples will be subjected to mechanical testing, which will be followed by evaluating important mechanical properties and critical parameters of coating material.

Key words: thermal barrier coatings, combustion chamber, atmospheric plasma spraying, mechanical testing, bend test, delamination

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Abstrakt

Termální nástřiky jsou rychle rostoucí postřiková technika v oblasti výroby používaná např.: v letectví, chemickém a petrochemickém průmyslu, automobilovém průmyslu, medicíně a biomedicíně. Mezi termálně stříkané povlaky patří termální bariérové nástřiky, což jsou žáruvzdorné vrstvy chránící základní materiál součástí pracujících v extrémně vysokých teplotách. Typickými výrobky používajícími termální bariérové nástřiky jsou turbínové lopatky, součásti strojů, ale také protézy. TBC poskytují ochranu před teplem, opotřebením a korozí, a proto se celosvětově používají pro široké spektrum aplikací.

Cílem této bakalářské práce je poskytnout obecné informace o termálních bariérových nástřicích, a postupně vyřešit několik otázek. Na začátku si představíme procesy a principy používané pro TBC nástřiky. Poté se přesuneme v rešerši na část zabývající se hledáním nejlepší varianty termálního bariérového nástřiku pro použití v letectví, konkrétně ve vnitřní části plamence ve spalovací komoře leteckého motoru. Bakalářská práce je psaná ve spolupráci s GE Aviation Czech, kde nám poskytnou vzorky z vnitřního plamence leteckého motoru potřebné pro testovací část práce. V experimentální části práce budou vzorky podrobeny mechanickému testování, po kterém bude následovat vyhodnocení důležitých mechanických vlastností povlakového materiálu.

Klíčová slova: termální bariérové nástřiky, spalovací komora, atmosférické plasmové povlakování, mechanické testování, test ohybem, delaminace

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Acronyms

APS	Atmospheric Plasma Spray
BC	Bond Coat
DC	Direct current
EB-PVD	Electron Beam Physical Vapor Deposition
HVOF	High-Velocity Oxy-Fuel
TATs	Tensile Adhesion Tests
TBC	Thermal Barrier Coating
TC	Top Coat
TGO	Thermally Grown Oxide

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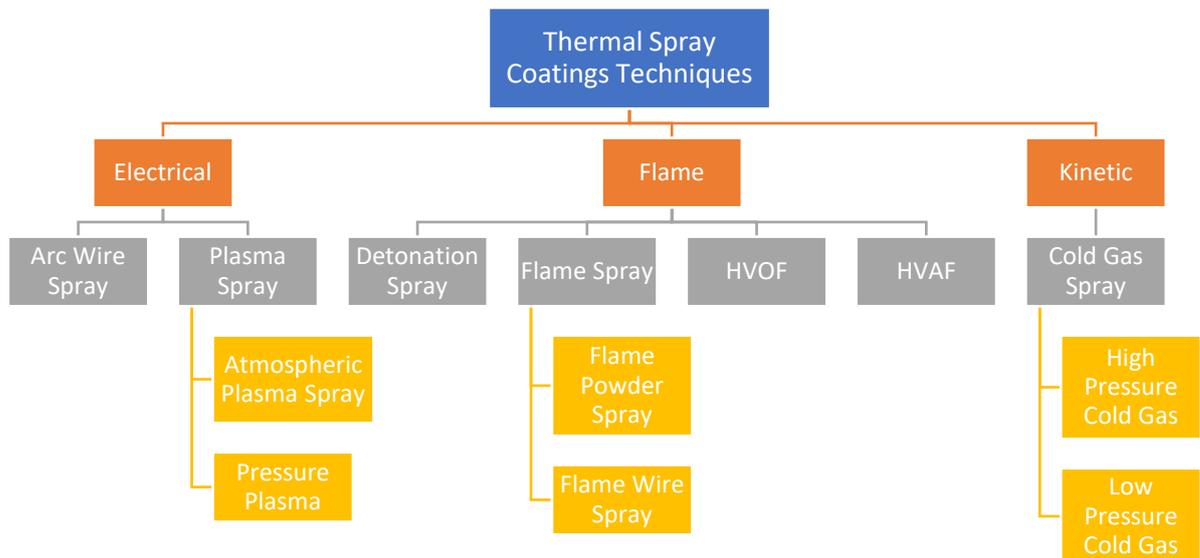
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Chapter 1

Thermal spray coatings

1.1 Introduction

Thermal spray coatings are spraying processes and coating techniques during which heated or melted materials or particles are sprayed onto a surface to provide usually very thick coatings (range of thickness is 20 μm to several millimetres whereby this thickness is depending on type of feedstock and material and used coating process). The purpose of the thermal spray coating techniques is solving problems of corrosion and thermal degradation. This coating process is very attractive, because it offers a wide choice of materials which have a reduced impact on the environment when we compare it to conventional plating processes. [2]



Tab. 1: Division of Thermal Spray Coating Techniques according to different methods. Information taken from [2]

From the table we can see that the Thermal Spray Coating Techniques are classified into different groups based on:

- Spray material used in feed stock of the spray gun
- Produced Kinetic Energy
- The Source of Thermal Energy

The coating materials are possible to apply on surface by using several different processes and methods. The application can be provided manually or with software-driven robotics with automated precision. The coating process can be performed in different environments. Materials can be applied in standard atmospheric conditions or in

specialized environment which must be highly controlled. To specialized environments, we include for example water. [2]

1.2 The principle of coating formation

The particles of melted additional material which is supplied in the form of wire, powder or hollow tubes fall on the surface. After melted material is spread over the surface area, instantaneous solidification occurs. Whole progress can be divided into four main parts:

1. Material enters into the energy source
2. Melting of particles of material
3. Melted material is transmitted on trajectory to surface
4. Cooling of coating on surface

Every phase has big impact on structure, quality and mechanical characters of coating.

The most important parameters for Thermal Spray Coating processes are velocity of melted particles and temperature. Both parameters are affected by technological options of the chosen method and process and partly also by the type of added melted material. The source of heat is utilized completely or partly, and its purpose is to melt added material and then transfer melted particles in the direction of the surface. [3]

Technology	Form of material	Source of heat [°C]	Velocity of particles [m·s⁻¹]	Materials	Character of coating
Electrical Arc Wire Spray	Wire	4,000	50 – 200	Metals, alloys, cermet	Bigger thickness, high density
Plasma Spray	Powder	12,000 – 20,000	100 – 800	Ceramics	Porous
Flame Spray	Powder or Wire	2,700 – 3,050	80 – 100	Metals, alloys, plastic	Bigger content of oxides and more porous
HVOF Spray	Powder	2,800 – 3,200	200 – 1,200	Metals, alloys, cermet	Big density, good adhesion, compressive stress
Cold Gas Spray	Powder	< 900	500 – 1,500	Soft metals and their alloys	Bigger thickness, small content of oxides

Tab. 2: Characteristics of Thermal Spray Coating Techniques. Information taken from [6].

Thermal Spray Coatings are part of nowadays technology for thermal barriers or insulation, abradable seals, dimensional restoration, wear protection and oxidation protection. The choice of spray technique depends on its application, availability of the process in trade and cost. [3]



Tab. 3: Technological parameters of the thermal spraying process influencing the resulting quality of coatings [7]

1.3 Substrate preparation and coating materials

The substrate preparation is the most critical step in the spraying operations. At first all surface contaminants including oil, paint and scale must be removed by brushing, acid pickling, scraping or vapor blasting or degreasing, because adhesion of coating is related to roughness and cleanliness of the surface. [3]

Surface of the substrate prepared for the plasma spraying is washed with the acetone liquid. To rough the surface, the process continues with blasting, for example by Al_2O_3 or SiC particles which leads to a better adhesion. [18] After this process the gas or air flow around the substrate must be controlled, especially during spraying to avoid the sticking of re-condensed vapours and debris. [3]

Blasting is followed by applying two coating layers to substrates. First layer is for instance a metallic bond coat and the second one is a ceramic top coat. [18]

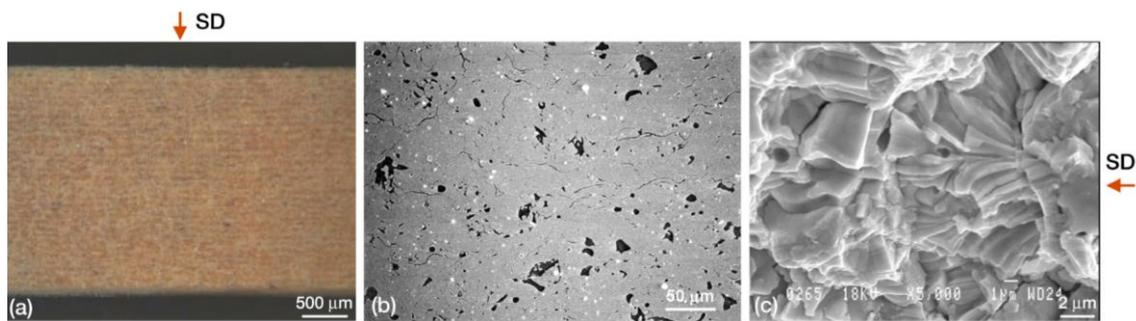


Fig. 1. Microstructures of plasma-sprayed ZrO_2 -8 wt% Y_2O_3 . “SD” indicates sprayed direction. (a) Side view of flexure substrate showing lamellae (layers). (b) Polished top surface. (c) Fracture surface [12]

Thermal barrier coatings have been developed since 1960s and mostly used materials for TBCs were TiO_2 , Al_2O_3 and $\text{CaO/MgO} + \text{ZrO}_2$. Materials selected for Thermal barrier coatings must follow requirements: high melting point, low thermal conductivity, chemical inertness, good adherence to substrate and low sintering rate of the porous structure. Nowadays typical commercial materials include $\text{CaO/MgO} + \text{ZrO}_2$ and 8YSZ used for top coat and NiCoCrAlY used for bond coat. [17]

1.4 Thermal spray coatings techniques

Thermal spray coatings are divided into three main sectors according to spraying method. These sectors are electrical spraying technique, flame spray technique and kinetic spray technique. Now we will provide information about particular spraying methods and determine what is typical for each one.

1.4.1 Electrical Coating Technique

Electric Coating Technique can be realized by one of two methods: Arc Wire Spray and Plasma Spray, which has two representatives, and these are Controlled Atmosphere Plasma and Low-Pressure Plasma (sometimes called Vacuum Plasma Spraying).

1.4.1.1 Arc Wire Spray

In this form of thermal spray coating method is used wire as a feed stock. Two current carrying wires are utilized in an electric arc to become the source to provide the heat. Between two automatically advanced, consumable wires short circuits of electric current which leads to creating a very high temperature of around 4,000°C. The tips of the wires start to melt which is caused by this temperature and once they are melted, inert gas or compressed air is utilized to atomize and accelerate the metal towards the surface of the substrate. Such process is ordinary used to apply coating to large areas to provide corrosion resistance on big components or for the building up of worn components. [2], [3], [4]

Typical coatings include copper and its alloys, nickel-based alloys, iron-based alloys, aluminium and zinc. The main purpose of Arc Wire Spray Technique is to provide corrosion protection, dimensional restoration and wear resistance. The advantage of this method is that different wires can be used to produce a pseudo alloy. Other benefits include:

- Substrates can be made of various types of materials
- Minimal facilities required
- With this method can be coated large areas
- High production spray rates
- It is not required to use combustible gas
- Excellent coating bond strength

In Arc Wire Spray process and Plasma Spray process, the substrate (which can be ceramics, composite, metal, plastic) has temperature far below its melting temperature. If surface is sprayed in air and substrates and coatings are modified at temperature range from 200°C to 500°C, it may lead to surface oxidation of the hot particles and coatings. However, if spraying takes place in a controlled atmosphere or in a soft vacuum with pressure range from 20kPa to 30kPa, oxidation is reduced. [3], [4], [14], [15], [16]

1.4.1.2 Plasma Spray

Nowadays there is a wide range of generating devices for plasma. Direct current (DC) transferred arc and DC torch, radio frequency inductively coupled torches and their hybrid combinations are primarily used, while DC torches cover the majority of the market (99%) and the most commonly 95% of them are working with guns at atmospheric pressure in air. [8]

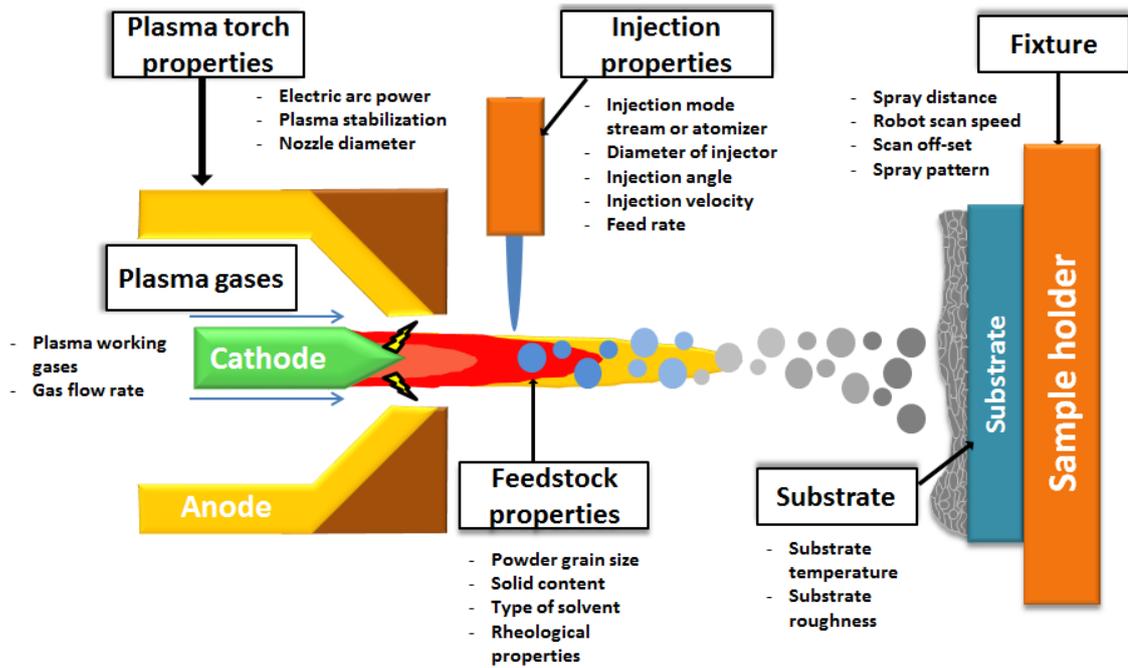


Fig. 2. Process parameters in SPS technique [11]

A typical plasma-spraying system generate plasma jet by a DC electric arc between 2 electrodes. Gas (mostly He, Ar and N₂) flows between the electrodes and its temperature range from 6 000 to 10 000 K. The gas is partially ionized by the electric arc to form the plasma. Form of material in feedstock is typically powder, but also sometimes is used wire, suspension or liquid. Material is injected in plasma and instantly melted. The molten particles accelerate towards the substrate where liquid droplets are flattening and create lamellae with lateral dimension from tens to hundreds of micrometres. [8]

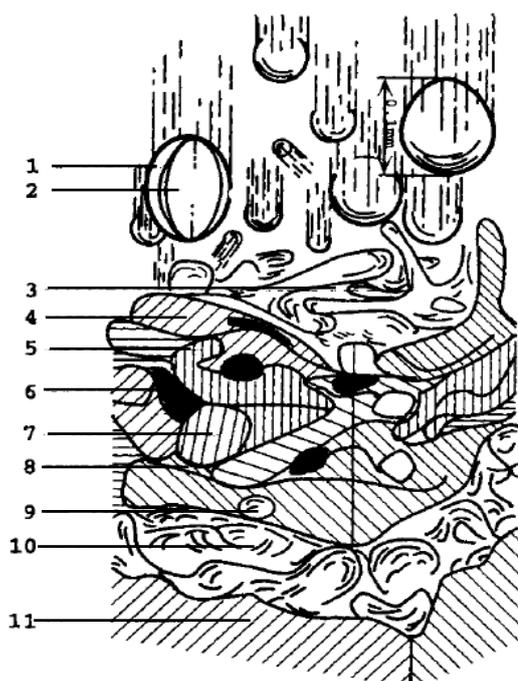


Fig. 3. Schematic rendering of structure of a coating layer. Picture and description taken from [1]

1. Molten shell
2. Unmelted core
3. Liquid splash
4. Lamellae also called 'pancake' splat
5. Interlocked splat
6. Oxidized particle
7. Unmelted particle
8. Pore
9. Void
10. Substrate surface
11. Substrate

1.4.2 Flame

Flame spraying technique is the first spraying technique developed at the beginning of the 20th century and which contributed to the development of the spraying technologies and methods. This method is divided into two principles and these are Flame Powder Spraying and Flame Wire Spraying. For this process are very important parameters like flame temperature which is typically in the range from 3000 to 3350 K and flame velocity which is kept at about 90m/s. Technique is carried out in air and the spray distance must be between 120 to 250 mm. Significant Principal Processing Parameter is substrate surface temperature. If we spray powder from ceramics onto metals, then surface temperature should be kept around 400 K to avoid the residual stress. [4]

1.4.2.1 Flame Powder Spray

Flame spraying of a powder begins with combustion of the working gases like fuel in oxygen where its chemical energy is used to generate a hot flame. Powder is injected to the torch and particles starts to melt in the flame, then the stream of particles goes through combustion flame and is accelerated in the direction of the surface of the substrate. Important process parameters for Flame Powder Spray are chemical properties which including crystal phases, chemical composition and physical properties like melting point and pressure of vapours. Material used for powder can be alloys, ceramics and metals but polymers is also possible to utilize for spraying. Particles size is typically in the range from 5 to 100 μm . In old types of torches powder feeders were gravitational and nowadays in modern types is used rotating plate feeding. [4]

1.4.2.2 Flame Wire Spray

As in the previous method a flame is produced by combustion of fuel gas. Flame spraying equipment for Wire Spraying is more complicated compare to equipment for Powder Spraying. [4]

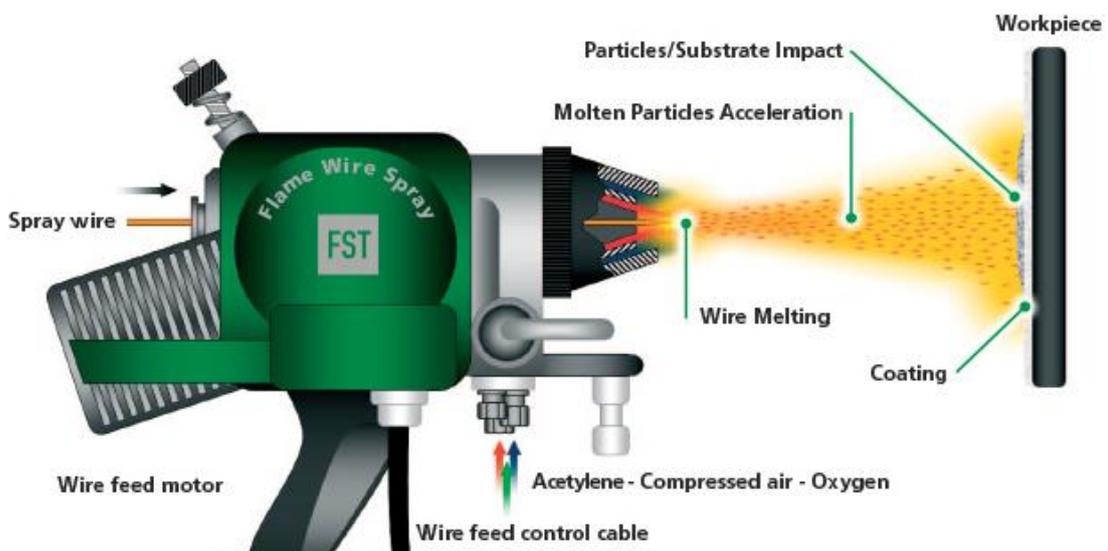


Fig. 4. A Spray Gun for Flame Wire Spray method [2]

The flame is generated in the compressed-air cap where oxygen, fuel gas, compressed air and working gases make mixture together. The end of the rod is melted by flame and then compressed air helps to atomize melted wire to create droplets, which shape a stream of molten droplets and are actuated towards the surface. For chemical composition of rods is utilized ceramics, such as, $\text{Al}_2\text{O}_3 + \text{TiO}_2$, Cr_2O_3 , and Al_2O_3 and for wires are used metals, such as, alloys of Zn, Mo and Al and stainless steel. The diameter of wires and rods is in the range from 3 to 6 mm, whereas factories frequently use diameters, such as, 3.17 or 4.75 mm. [4]

1.4.2.3 HVOF

The High Velocity Oxy-Fuel Spraying is coating technique which increases wear resistance mainly for large parts. By 2000 HVOF took over one quarter of the thermal spray market, because the HVOF metal coatings met the characteristics of Plasma Spray Coatings at a lower cost. Spray process of the basic operating principle is very simple. Hot gases are produced in high pressure combustion and used for heating and accelerating material in a powder form with typical diameter 20-80 μm . [8], [9] The fuel and oxygen expand and are accelerated through a nozzle, which leads the gas to reach high speed to 2,200 m/sec. The powder is injected in the gas flow. If the injection is made axially, the flow reaches very high speed up to 800 m/s and during radial injection it leads to extremely high speed around 2,200 m/s. The flame temperature is depended on the selected sprayed fuel and reaches values in the range from 3,000°C to 4,000°C. Materials utilized for the HVOF coatings include carbides (chromium carbide, tungsten carbide), cermets, super alloys (typically based on nickel, iron or/and cobalt) and MCrAlYs bond coatings under Thermal Barrier Coatings. The HVOF coatings ensure high bond strength, low porous, low degree of oxidation, cavitation protection (typically used in hydroelectric power station) and one of the best corrosion properties (HVOF are utilized for their good protection against chemical corrosion, for example in impellers and pump casings). [2], [4], [9], [10]

1.5 Summary of Chapter 1

Based on qualitative analysis of resulting properties of coatings and intention of utilisation, it concludes that the possibility of using high temperatures in source, oxidation behaviour and resulting microstructure are important factors when choosing thermal barrier coating technique. Therefore, this research is focused on thermal barrier coatings suitable for use in combustion chamber of an aircraft engine. The properties of individual methods indicate that plasma spray coatings are the most appropriate coatings for our need in aerospace industry.

Chapter 2

Plasma

2.1 Introduction

The advances in plasma spray coating technique in previous part led to the conclusion that this is the most suitable method for our experiment. Now an overview must be given to find out what plasma is and what are its applications to confirm its method is advantageous for utilization in the combustion chamber of an aircraft engine.

2.2 Basic definition of Plasma

Plasma is considered as the fourth state of matter and more precisely a quasi-neutral multiparticle system meaning plasma's charge is neutral even though it is electrically conductive. [1] Plasma state is achieved by transferring energy into a gas. Ions and electrons are allowed to act independently once there is enough energy to ionize the gas. Plasma is produced in an electric field when currents become the free electrons able to move in ionized gas. After the energy source is removed, both ions and electrons create heat and energy by mixing with each other. Electric arc and induction-coupled plasma discharges are energy sources for plasma. In a DC plasma arc, core plasma temperature is over 20,000°C.

Plasma-sprayed coatings compared to electric arc spray and flame coatings is the leading spraying technology due to its high degree of melting particles, high velocity of particles, high deposit density and high bond strength. The porosity of coatings is as low as porosity of HVOF sprayed coatings depending on configuration, gun type and properties of material. The porosity is a function of the characteristic parameters of the gun and the powder and may range from less than 1% to 40%. The high quality of the thermal barrier coatings made by plasma results from the high kinetic energy of particles and droplets. Coating bond strength is getting between 34 to 69 MPa. [21]

2.3 Fields of Application of Plasma Spraying

Plasma-sprayed ceramics and metal coatings have a wide range of application, mostly in aerospace and aviation, agriculture, automotive, engineering, mining, medical devices and biomedicine. Users demand to protect their equipment and machinery from corrosion, chemical and thermal attack and wear and require new properties such as bioceramics and high temperature superconducting coatings.

Coatings have been used in many industrial areas. Examples of specific applications are listed below:

- Chemical and thermal barrier coatings are mostly used for combustion cans and gas turbine blades in aerospace to prevent hot gas corrosion, and in adiabatic diesel engine for valves and pistons

- Wear control is required for bearings, printing rolls, valves, pump plunges and turbine vanes and machinery parts in coal power plants; also, in high-power electronic chips where diamond coatings are used for wear control
- Corrosion protection has demands in chemical and petrochemical plants, internal combustion engines and agricultural applications to protect floors from fruit juices and operations in dairy industry
- Dental and orthopedic prostheses requiring biocompatible properties [1]

2.4 Applications of Plasma Spraying in Aviation

In 1911, after several patents, M. U. Schoop began utilizing lead coatings to improve corrosion protection. The field started to develop quickly and in late 1950s plasmatrons helped significantly to boost development. The D-gun coatings found applications in the aerospace and main technological growth began due to coatings based on stabilized zirconia. After 1950s till the 1980s TBS's ceramics had spread in aircraft engine coatings. The second wave of growth started by low pressure - vacuum plasma spraying in the 1980s which found purpose in aerospace gas turbines.

Large importance and impact in the aerospace industry had development of coatings based on yttria partially stabilized zirconia which increased the lifetime of components, such as austenitic superalloys blades in gas turbine engines, turbine shrouds and combustor cans. An uncoated component parts have limited lifetime by hot corrosion and thermal and mechanical fatigue around 800°C. Plasma-sprayed coatings help to prevent hot corrosion caused by gas and salts and increase cooling efficiency and that makes transatlantic flights possible. [1]

2.5 Summary of Chapter 2

In our research plasma spray samples from combustion chamber of an aircraft engine will be used for following experimental part. Currently, there is no better coating process for use in aerospace and only plasma spray process is improving. The resulting properties and the approach of plasma spray coatings provides new insight into future changes. The efficiency of gas turbine depends on inlet temperature and the value of temperature is slowly raising due development and plasma spraying solve such problem partially. Compare to conventional coatings, microstructure produced by plasma spraying is finer but problems such as erosion and thermal shock resistance are not sufficiently improved. [11]

Chapter 3

Mechanical testing

3.1 Introduction

Mechanical test methods have been developed to describe mechanical properties of materials. These are necessary to know for the manufacture of any product. Methods covers a large number of tests and a wide range of them are standardized. Standards are for instance in ASTM which are recognized worldwide.

Types of mechanical tests:

- Dynamic – the force suddenly increases to a certain magnitude during the test.
- Fatigue – cyclic loading is applied on structure to collect data and identify critical location. Depending on the character of the emerging stress, the fatigue loading is divided into three types: alternating load, pulsating load and repeated load.
- Static – the tested sample is subjected to a slowly increasing force up to a permanent value. Static tests include tension test, bend test, pressure test, torsion test and shear test.

Mechanical testing of thermal spray coatings determines mechanical properties, such as wear resistance, hardness, bond strength, corrosion resistance and high temperature properties. Nondestructive and destructive tests are used to assess residual stress in thermal barrier coatings. The chapter will describe several mechanical tests used for thermal barrier coatings.

3.2 Thermal Fatigue Testing

Thermal Fatigue Testing is utilized to determine thermomechanical stability. The sample is exposed to a thermal cycle and examined with microscope for spallation-type cracks. The thermal cycle is carried out by rapid heating and rapid cooling rates using heating jets, with short time holding at the maximum temperature. This test is challenging for the oxide layer, because the bond coat persists at low temperature due zirconia layer nature and little time at high temperature. [21]

3.3 Fracture Mechanic Toughness Testing

Fracture Toughness Test investigates interface fracture mechanics theory and determine fracture resistance of multi-layered TBCs. A failure of thermal Barrier Coatings initiates from internal defects or voids and the Fracture Toughness Test evaluate a crack inside the coating. The method has two stages. The first stage is computational crack analysis. A reference tensile load is applied to the crack with circular shape in a perpendicular

direction. Mixed-mode stress intensity factors and energy release rate are characterizing parameters used for determination of critical fracture toughness. In the second stage experiments are carried out and measure a critical tensile load needed for failure. Reference factors and critical load found in the first stage are utilized to determine the critical fracture parameters. [15]

3.4. Impact Testing

The Impact Testing determine fatigue strength and the dynamic creep behaviour of coating. The coating is under a maximum impact load and during the test, a carbide ball periodically penetrates the substrate. In order to determine creep behaviour, also known as dynamic time-dependent plasticity, several impact loads imprints must be performed. [19]

3.5. Tensile Adhesion Testing

Tensile Adhesion Tests (TATs) measure the bond strength of coatings and examine failure mechanisms of TBCs, mainly plasma sprayed coatings. A test pull stub (dolly) is glued to the coated surface and is exerting a force to the surface in a perpendicular direction to remove both the pull stub and the coating from the substrate. The purpose of tensile adhesion testing is to measure the force at which the coatings fail and to obtain the type of failure. [16]

3.6. Bend Test

The mechanical behaviour of Thermal Barrier Coating sample can be obtained using the Three Points or Four Point Bending Test. These tests are used to study crack initiation and damage evolution under mechanical loading at room temperature. The Three Point Bending Test is performed on a testing facility, where the device has two main parts – a punch and support rods. During the test, the inhomogeneous distribution of stress occurs throughout the length of the sample and at the central loading position the stress reaches a local maximum. Both Conventional standard uniaxial Three- and Four-Point Bending Tests are mainly used to evaluate the interfacial properties of brittle materials which include Thermal Barrier Coatings and to investigate the delamination resistance. [14]

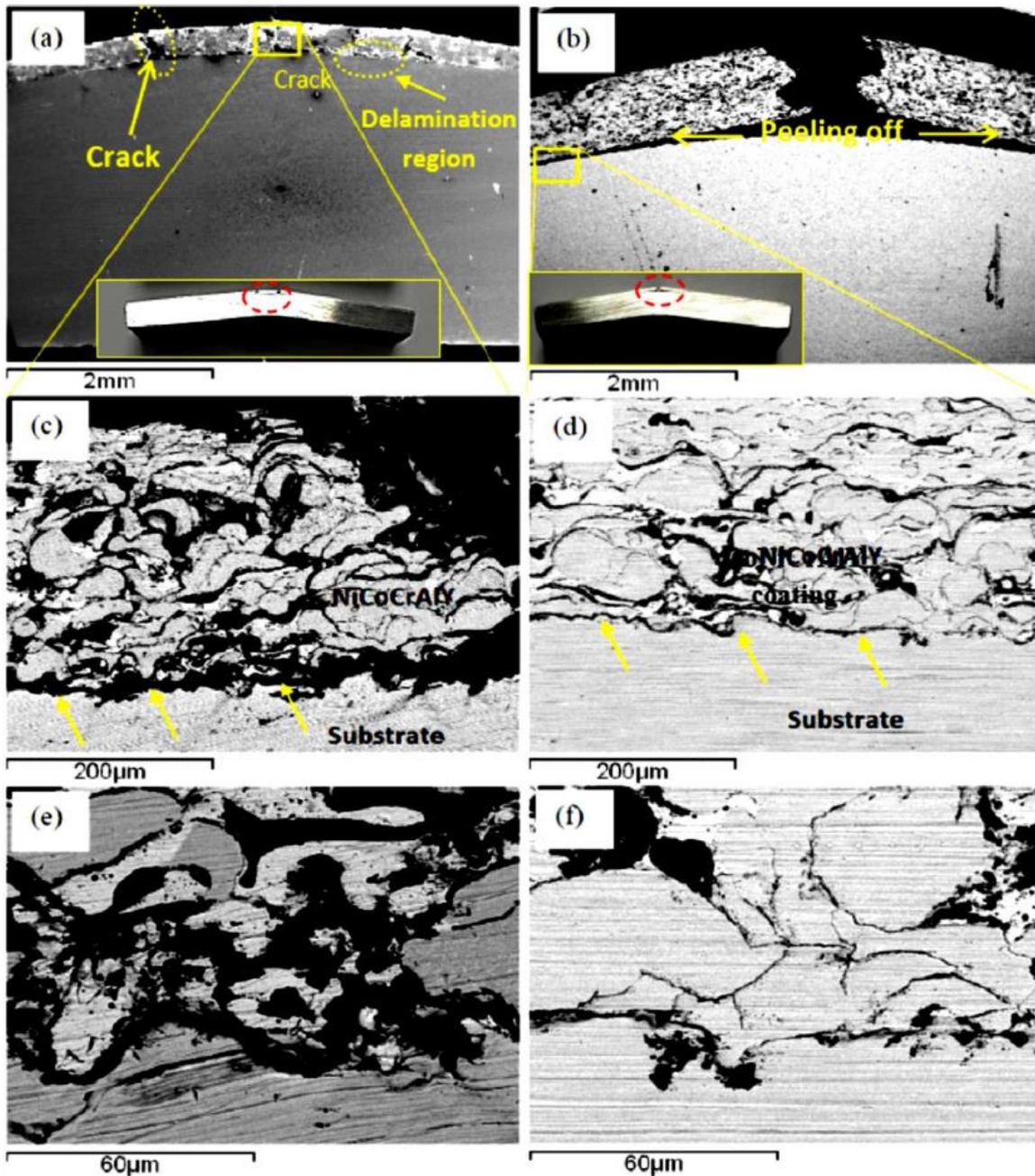


Fig. 5. The fracture micrographs and macro images of coatings on stainless steel after the Three Point Bend Test. Arrows indicate the crack on substrate interface. [21]

3.7. Summary of Chapter 3

The studies we reviewed employ various methodologies in order to collect data about mechanical properties of tested subject. For our needs we choose to realize only one mechanical test, which is the most suitable for experiment to find the mechanical properties of plasma sprayed coatings. Out of number of effective testing methods in my experiment I would like to conduct mechanical bend test as part of GE standard requirements utilized to find critical parameters and values affecting crack initiation and damage evolution under loading.

Chapter 4

Design of the Experiment

4.1. Historical Introduction and Motivation

Thermal Barrier Coatings have been the subject of many studies in Material Science and Aerospace Engineering and an important object of research since the 1960s. TBCs are of interest because as we stated in previous chapters their main purpose is to insulate combustion chamber in the engine and turbine components from the hot steam of gas as well as to improve durability of components and its energy efficiency. [22]

There is a growing body of research that successfully accept TBCs in the combustion chamber in aircrafts as a key factor in protecting the hot structures of turbine engines. TBCs play a pivotal role in worldwide research focused on the increasing efficiency of gas turbine technology. [24] Early examples of research include the first successful test conducted in the turbine section in the middle 1970s. Research began at the National Aeronautics and Space Administration's NASA Lewis Research Centre. Plasma sprayed NiCrAlY created the bond coat and a porous zirconia-yttria ($ZrO_2-Y_2O_3$) ceramic sprayed by atmospheric pressure plasma were used as top coat. The first entry on the vane platforms in gas turbine engine emerged during the 1980s. Later in studies was discovered another type of deposition process named physical vapor deposition. Both types are currently utilized in commercial aircraft turbine applications. [23] However, APS is more economical, and trend is growing towards the use of atmospheric plasma spraying. [24]

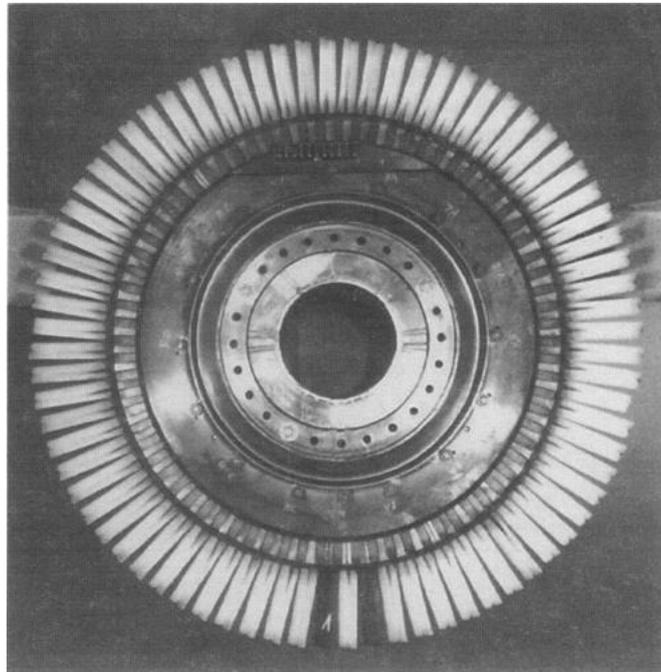


Fig. 6. Turbine J-75 blades with coating $ZrO_2-12Y_2O_3/NiCrAlY$ after completing the testing of aircraft part. This test was significant for the beginning of the modern era of Thermal Barrier Coatings. [23]



Fig. 7. X-15 rocket plane had on the exhaust nozzle coating made of Zirconia-calcia/NiCr which is the first use of Thermal Barrier Coating in flight with crew. [23]

Plasma Sprayed Coatings on substrates as well as other TBCs are comprised of a bond coat, TGO (Thermally grown oxide) layer and top coat which together provides thermal protection. One of the main obstacles to the proper functioning is the delamination which is the expected failure mechanism occurring usually between the bond coat and the TGO. The demanding operating conditions could lead to such problems during service and previous experiments have revealed that delamination between layers is occurred by the initiation and progression of microcracks. Proposed experimental methods for examination of delamination and its features are pull off test, tensile test, bend test and shearing test. [22] In the Experimental Part of this Bachelor Thesis we will determine the Bend Test as a major topic of interest within the field of representatives in mechanical testing of TBCs.

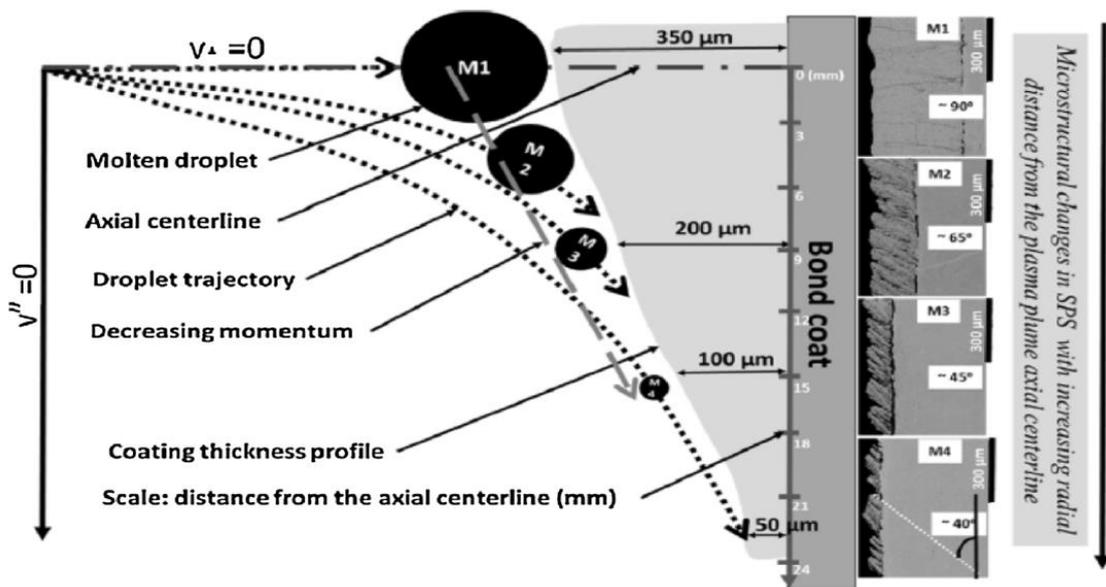


Fig. 8. The figure shows how the droplet trajectories have different results in the microstructure of the coating due to their differences in length. [24]

4.2. Problem statement

There are now sufficient researches on the benefits of TBCs with main focus on Plasma sprayed coatings. A lot of research has been devoted toward the need for creating coatings which are able to operate under demanding conditions. However, not many researches have been reported on how to implement tests in order to examine mechanical properties of coatings and eventually continue in research to enhance properties of coatings and slightly improve future results of tests.

The aim of this research is to carry out experiment concretely bend test which will help us to evaluate delamination and microstructure of samples taken from new component of aircraft engine and samples of already used engine. During the experiment quantitative methods will be used to identify changes in microstructure in individual samples. Then it will be followed by using statistical analysis to compare results of individual tests and evaluate dependency or analogy to expected normalized and standardized results.

Nowadays the manufacturing process for products using Plasma sprayed coatings is as efficient as possible. If results of tests come out as unsatisfactory and possible incremental loss in efficiency would occur that would mean the interest in other types of Thermal Barrier Coatings can grow and companies could find new solution to meet product goals.

4.3. Significance of the study

GE Aviation Czech, as an operating unit of the General Electric conglomerate, is one of the top aircraft engine suppliers which offers mainly jet and turboprop engines and other components for commercial aircrafts, but also for business and military aviation.

The Bachelor Thesis was written in cooperation with GE Aviation Czech. The company, figuring in thesis as task submitter, proposed research topic in Thermal Barrier Coatings with its target to determine what is the best option for mechanical testing of TBCs, mainly atmospheric plasma coatings significantly used in aerospace to find out how to avoid most of the unwanted delamination which can occur in worn parts but also in primary production.

The aim of the present research is to examine delamination of 4 used samples and 1 new sample provided by GE Aviation Czech to find out what are their possibilities of mechanical testing and mainly which failure modes of coatings can occur during primary production or utilization and which failure mode is the most preferred all within the GE Aviation Czech standards of measurement with specified criteria. The substrate is made of material with trade name Nimonic 80a. Nimonic is nickel-chromium alloy with additions of aluminium, titanium and carbon. This alloy consists of 1 – 1.8 % of aluminium and 1.8 – 2.7 % of titan. [31] Coating material is yttrium stabilized zirconia oxide. Temperature in laboratory was 17°C during the process of the testing. There will be 4 samples already used for 3000 flight hours in flue gas environment in the engine. The reason why we test sample after 3000 flight hours is that it is the time when the first general inspection takes place. Unfortunately, we couldn't test more samples for this thesis because of coronavirus pandemic which prevented us from accessing laboratories at the university and in GE Aviation Czech.

Taken together, outcome condition of the sample after the bend test provides important insights into condition of the coating. As a promising result we can expect sample with cracks where coating stay attached to substrate. In this case, we can evaluate loss of continuity between lamellas which usually happen between two phases. If coating peels off of substrate then based on criteria and standard results, we can see the sample as unsatisfactory and deal with the question: when the defect of sample occurred – if in primary production or after utilization, which could follow other more detailed questions.

Main causes of failures of coatings are improperly prepared surface, inadequate specification and defective coating. We can divide failure modes into categories: formula-related failures, physical defect-related failures and substrate-related failures. The most important in the case of TBCs are substrate-related failures as interface contamination and physical defect-related failures as voids and delamination. The contamination can be caused by corrosion products and after abrasive blast and lead to rust, blistering and loss of adhesion. While cause of voids is regardless of the preparation method if the deposition process involves phase transformations like excessive evaporation and condensation, microscopic voids are formed by the thermal motions in the liquid and can generate the nuclei which rupture, grow and create bubbles. [29]

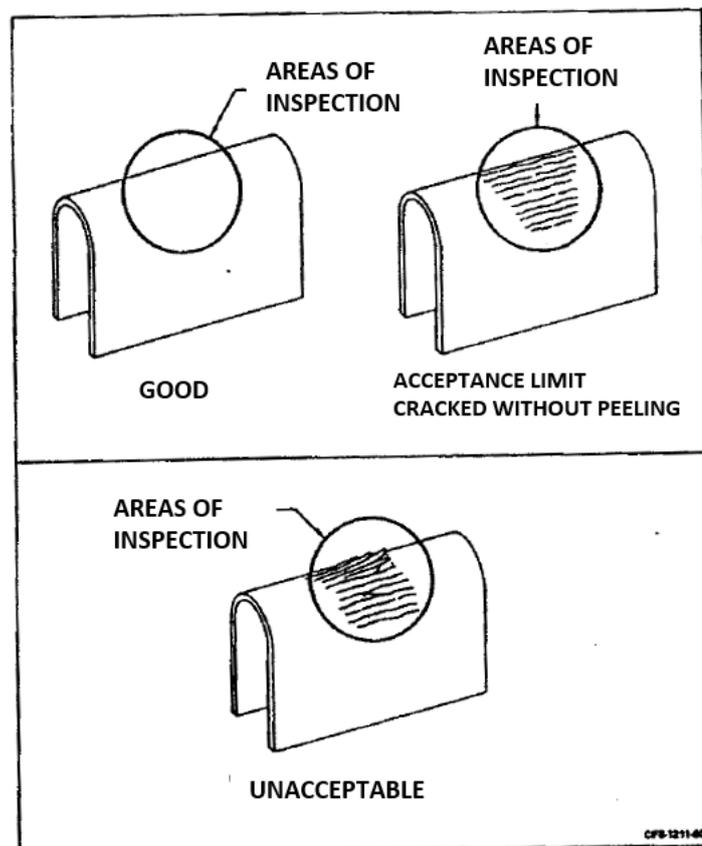


Fig. 9. Acceptance for bend test of sample for testing method based on GE Aviation Czech criteria and standards. As you can see from the picture, peeled off coating from substrate is unacceptable result after bending test. Only acceptable result is sample with attached coating with cracks and microcracks.

Delamination is defined as a failure mode of coating adhesion to underlying surface or between layers of coating where separation occurs along a plane parallel to the coating layer or a part's surface. It is expected that delamination does not occur often while in use in aerospace industry where safety is very important factor. However, a major problem is that the performance of coatings is limited by several circumstances, mainly by weak bonding of coating to underlying surface so in that case delamination is dangerous kind of failure as it can develop inside of the material, without being first visible on the top layer of coating. While some research has been carried out on delamination and its problems in coatings, only few studies have attempted to investigate different causes of delamination in TBCs and there is little scientific understanding of processes which can help reduce or avoid delamination of TBCs' layers. [25]

As already said, delamination can be created after several flight hours of aircraft or during production of coating. In primary production, the problem starts with spraying of molted particles accelerating towards surface of component. Spraying is uncontrollable process within which one coating can have several empty spots of unmelted particles. When coating's thickness grows, unmelted particles can connect in certain places or in certain directions leading into creation of crack. Later, inner tension in coating caused by crack will develop into delamination.

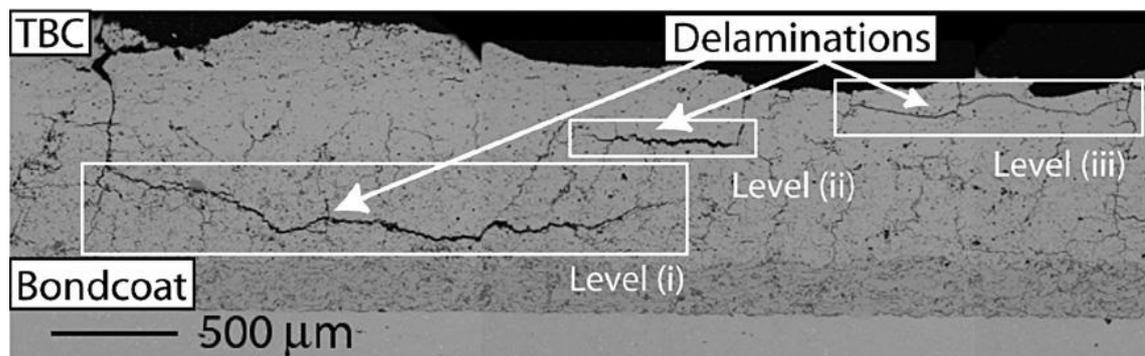


Fig. 10. Coating with thickness 1mm deposited by air plasma spray. [28]

This paper analyses mechanical characteristics important to predict delamination and assesses the significance of investigation of laminate thickness on interlaminar stress which help us to find out in which layer delamination grow more easily than other layers and in which layer is larger strain energy released. [25] The purpose of this thesis is to investigate the factors that determine the range of limits in testing and seeks to explain the development of delamination testing methods. Research reviews the data from three-point bend test, aiming to provide evidence how delamination occurs and behaves in atmospheric plasma sprayed components. My goal for this work is to provide new fresh insights into method used for delamination testing. However, this study is unable to encompass realization of experiments of the entire scale of possible testing methods. It is beyond the scope of this study to examine all of them in terms of writing time of thesis and it is not the task of this thesis.

4.4. Instruments

Over the past half century, a number of techniques for testing delamination of coatings have been developed. Nowadays most important methods include pull-off test, four-point bend test, three-point bend test, cross-cut test, etc., but there are also other methods for examination and detection of delamination being developed such as luminescence mapping. Different methods of testing and non-invasive detection techniques are focused on investigation of various properties and different delamination behavior. After my search for most common testing method for determining behavior and characteristics of delamination, I found out that in most recent studies, their authors used mainly four-point bend test. A major advantage in research of four-point bend test is that it's utilized so often that researcher can use a lot of data from previous studies and have opportunity to find more detailed outcomes with which they can focus on better improve of coating. Not just that the 4-point bend test is widely utilized to examine the adhesive and interface properties in thin film structures, it is also used to study the time dependent subcritical debonding of interconnect structures and to determine crack velocity in dependence of coating thickness and the fracture resistance. [26]

Since we have option to choose from many testing methods, I decided to propose three-point bend test. This research design has been used in few studies in the past to investigate the mechanical properties of TBCs, but there are very limited results. I would like to determine the factors that affect delamination behavior and gain a detailed understanding of method since a narrow range of previously reported papers were published. Method was selected for its reliability and validity in research sphere and its simplicity for implementation. During the 3-point bend test many critical cracks occur at the top-coat's surface. Also, some cracks may propagate towards the bond-coat interface or towards the substrate of sample. The failure mechanism of the Atmospheric Plasma Sprayed coating under the 3-point bend test is being caused by the debonding of coating from the substrate of the sample and also the propagation of the horizontal crack along the interface between the bond-coat and the substrate under the effect of flexural moment. [27]

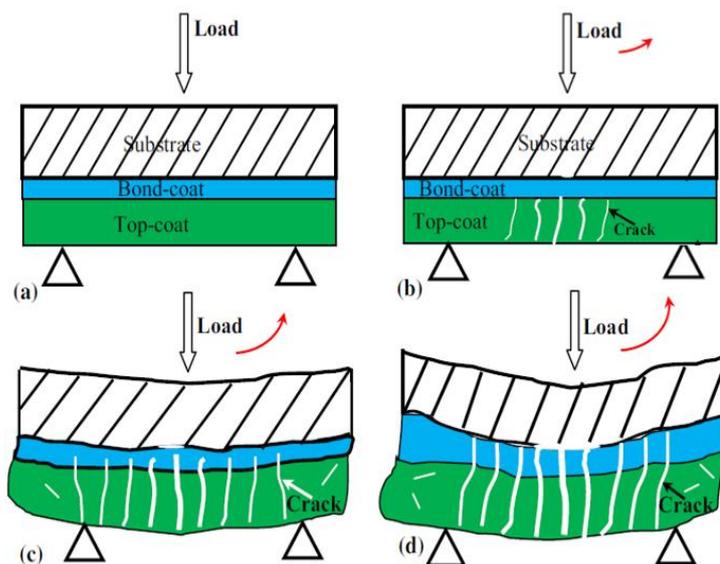


Fig. 11. Failure system of substrate yttrium stabilized zirconia oxide coating under the 3-point bend test. (a) Substrate without loading force. (b) After force starts to affect substrate and coating, microcracks propagate towards the coating interface. (c) Cracks pass through top coat and bond coat and reach to substrate interface. (d) The horizontal cracks initiate and propagate along the substrate and bond coat interface. [27]

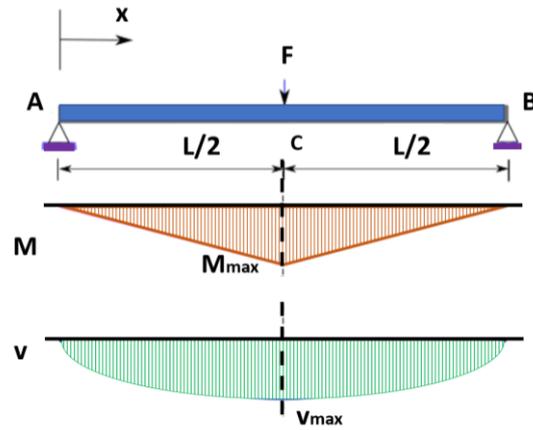


Fig. 12. The figure illustrates the three-point bend test. The loading force operate in the middle of sample which rests on two supporting pins. Under its schematic, graphs of moment M and deflection v .

After the bend test, we will receive measured data of the loading force and a deflection. Based on the schema on the figure 12, we can use acquired values to calculate a flexural strength while using calculation of a section modulus and a maximum bending moment defined as multiplication of a half maximum loading force and a half length of the sample. [32]

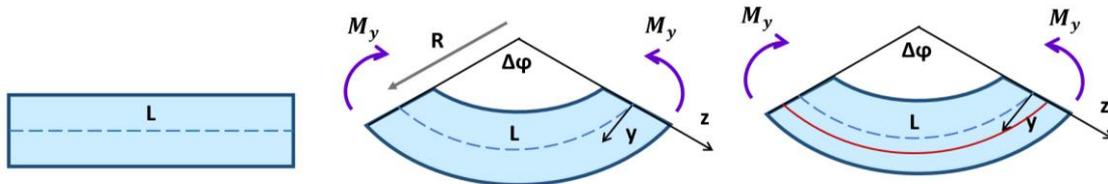


Fig. 13. Figure shows process of creating an evenly bend sample.

When we look at the figure 13, we can see that the original length of the unchanged prismatic beam midline is $L=R \cdot \Delta\varphi$ and the radius of curvature is R . We consider simple bend caused by the moment M_y which deforms the beam into the shape of a circular segment around the main central axis of inertia y . Elongation of fibre occurs and new length of fibre is $L+\Delta L$ where $\Delta L=z \cdot \Delta\varphi$ so $L+\Delta L=(R+z)\Delta\varphi$.

The beam can be seen as a system of fibres and transfer fibre deformations and transverse stresses are neglected. Along the height of the cross section there is a linear change in deformation which corresponds to a linear change in stress according to Hooke's Law

$$\sigma_x(z) = E \cdot \varepsilon_x(z)$$

where E is Young's modulus and $\varepsilon_x(z)$ is linear change of deformation. [32]



Fig. 14. Bernoulli-Euler Theory of bending stress distribution.

To analyse the behaviour of bending elements, we can utilize The Bernoulli-Euler Beam Theory. It focuses on the beams which are subjected to lateral load. When we take a look on the figure 13, the picture on the right, and compare it to the figure 14, we see that in the top part of the beam is created pressure, while under midline is thrust. Our sample consists of substrate and coating which has its parts – bond coat (BC) and top coat (TC). In the substrate and its upper part, bending stress creates compression. In the upper part of the substrate, bond coat and top coat tension occurs. Their values in each section, meaning in top coat and bond coat, are different. Tension in the BC can have prominent value in comparison to tension in substrate and TC. In conclusion, we need to notify that stress values change through the coating since it is consisting of two main parts – top coat and bond coat. Fibre contributes to the moment with value $z \cdot F(z) = z \cdot b \cdot \sigma_x(z)$ so the bending moment to the main central axis y is defined as

$$M_y = \int_{-\frac{h}{2}}^{\frac{h}{2}} z \cdot F(z) dz = \int_{-\frac{h}{2}}^{\frac{h}{2}} z \cdot b \cdot \sigma_x(z) dz$$

In simple bending, the bending moment is the only one internal effect which means that the resulting normal force on the neutral axis passing through the centre of gravity of the cross section is zero. Before deformation, original fibres are straight and after deformation, fibres are curved, the bottom fibres are extended, and upper fibres are shortened and between them is neutral axis whose fibres do not change length. [32]

The primary original data from the experiment will be collected directly by me. Such data manipulates and controls variables of experiment to determine cause and effect, because we are testing causal relationship between variables. The research method, for collecting and analyzing data, will be quantitative which is used to systematically describe collections of information and is expected to generate reproducible knowledge. For subsequent evaluation of the data, I will utilize statistical regression test which looks for the effect of variables on another variables. This methodology will be used according to prevailing evaluating method, which is an effective way of obtaining further in-depth information to answer research questions.

Chapter 5

The Course and Results of the Experiment

5.1. Data collection

5.1.1. Samples, Testing Equipment and Test procedure

Samples are acquired in GE Aviation Czech laboratory from combustion chamber of turboprop aircraft. The coating's thickness is $100\ \mu\text{m} = 0.1\ \text{mm}$ and the substrate's thickness is $1.7\ \text{mm}$. Samples have approximate dimensions of $24,5 \times 14,3 \times 1.8\ \text{mm}$.

Samples were prepared by cutting up a part of the combustion chamber of the aircraft. During a wet abrasive cutting process was utilized cutting equipment with a wet abrasive cutting machine in the GE Aviation Czech laboratory to take representative samples. The resulting samples can not be subjected to any damage which could modify material structure and at the same time. The cutting process has to be tailored to the chosen material and component's application.

The testing equipment used in the experiment include a Servo hydraulic testing frame ZUZ 200 designed for fatigue tests of material samples.

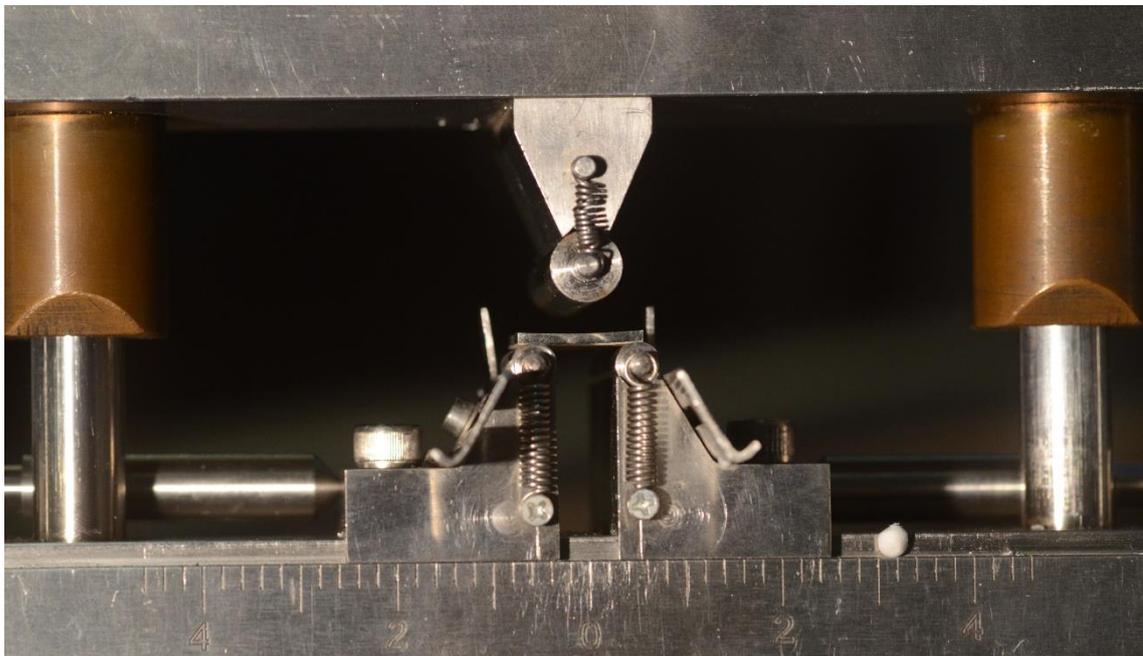


Fig. 15. Sample is laid in testing equipment ZUZ 200.

The function of the loading frame ZUZ 200 is to load the selected samples with coatings by determined deformation or force or position. The equipment can test tensile strength of samples and comprehensive strength and provide fatigue tests for different cyclic loads or frequency or amplitude and whole process is controlled by computer with software for

data recording. The diameter of the top cylinder in testing equipment ZUZ 200, which puts force on the sample, is 6 mm. The pitch, as the distance between the rollers on which the sample is placed, is 14 mm. Samples were loaded by force with constant speed 0.0166 mm/s till the end of test.

Once the test is completed, the structural properties of metal substrate and coating are analysed under a light microscope. Analysis is usually used to evaluate material and mechanical characteristics in the process stages of components, but also in cases during which damage occurs. The evaluation of samples in our testing method is based on few criteria: type of cracks, their size, distribution and orientation and the angle at which the sample bent.

After receiving samples, they were loaded in testing equipment to complete the 3-point bend delamination test under displacement control until a crack propagated on the top coat of the coating. Whole process was monitored and recorded on camera Nikon D 700.

5.1.2. Data presentation

Process of the testing with the use of servo hydraulic testing frame ZUZ 200 is controlled by a computer which records measured data in real time. Table 4 presents the experimental data measured on the sample 1 which was used in the combustion chamber with 3000 flight hours. Measured experimental data from all samples were recorded in the similar way.

Time	Position	Force 1	Force 2
[s]	[mm]	[kN]	[kN]
00:00:00.0000	-44.35853035	-0.001642505	-0.001893315
00:00:00.0200	-44.35791651	-0.002120942	-0.002096782
00:00:00.0400	-44.35740497	-0.002601155	-0.002539865
00:00:00.0600	-44.35760959	-0.004281901	-0.004036497
...

Tab. 4. Example of a few measured experimental data from the sample 1.

Table 4 shows the dependence of the position of the centre of the sample on time and the force. The force was measured by two piezoelectric sensors which are used for measuring dynamic forces and helped us to make sure that the measuring is going correctly.

Data of each individual testing process were saved to a csv (comma separated values) file in MS Excel while the set of measured values of one test contained around 77 000 lines. Values from measuring were time, force, position, which are detected by sensors. Values of force and deflection are the output of the measuring recorded in a digital form.

After completing mechanical testing of all samples, we moved back to the GE Aviation Czech laboratory where we put them under the light microscope to provide analysis of microstructure. On the following figures, you can see cracks in microstructure of samples which cause delamination of coating and the evaluation of the results of the bend test.

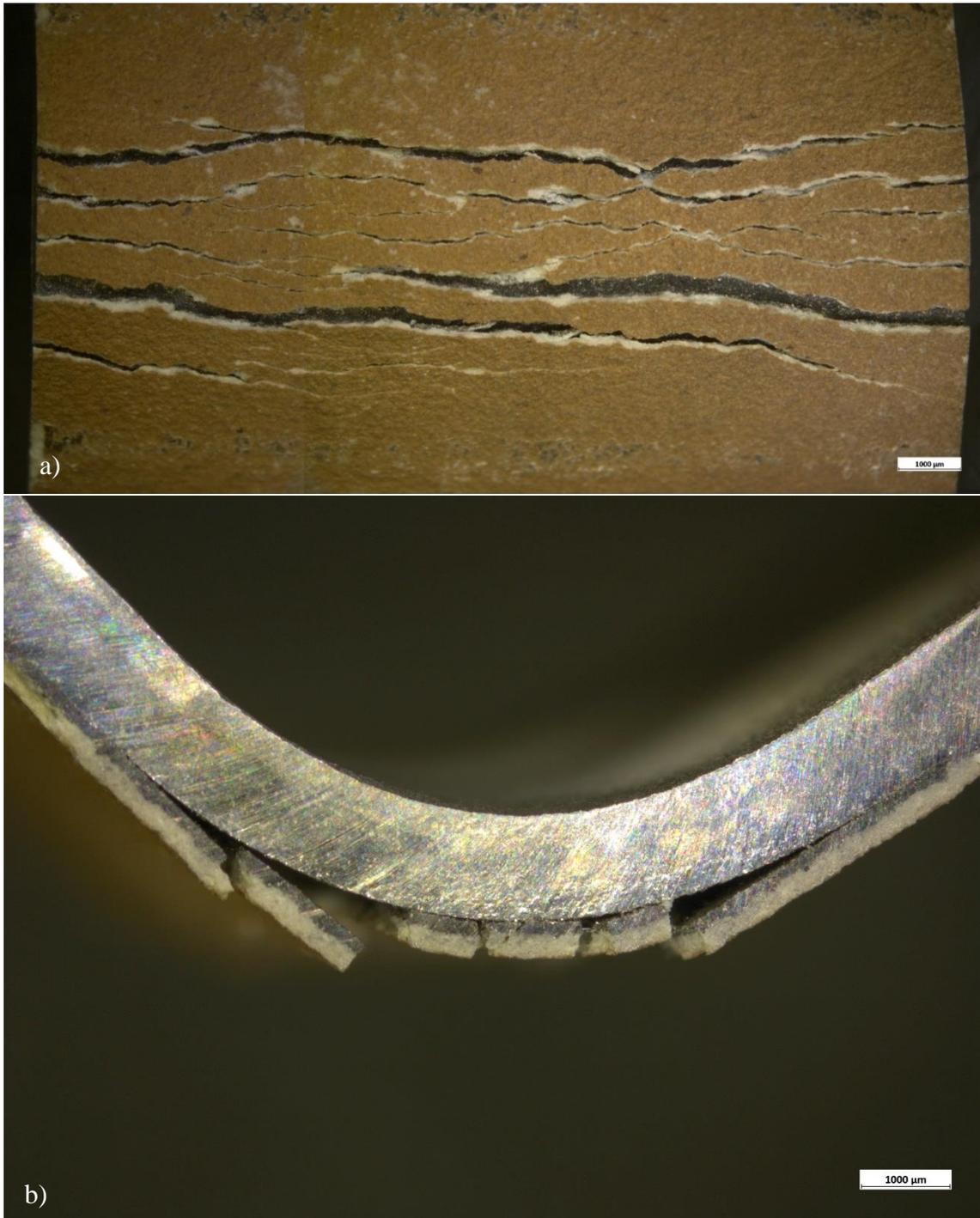


Fig. 16. Microstructure of the sample 1 from used combustion chamber of aircraft after 3000 flight hours tested on delamination.

What stands out in the bottom picture of the figure 16 is the peel off effect of the coating. Referring to GE standard showed in the figure 9, this result after bend test is unacceptable. In further investigation, we will solve what the cause of this result is and how this result is viewed during the general inspection.

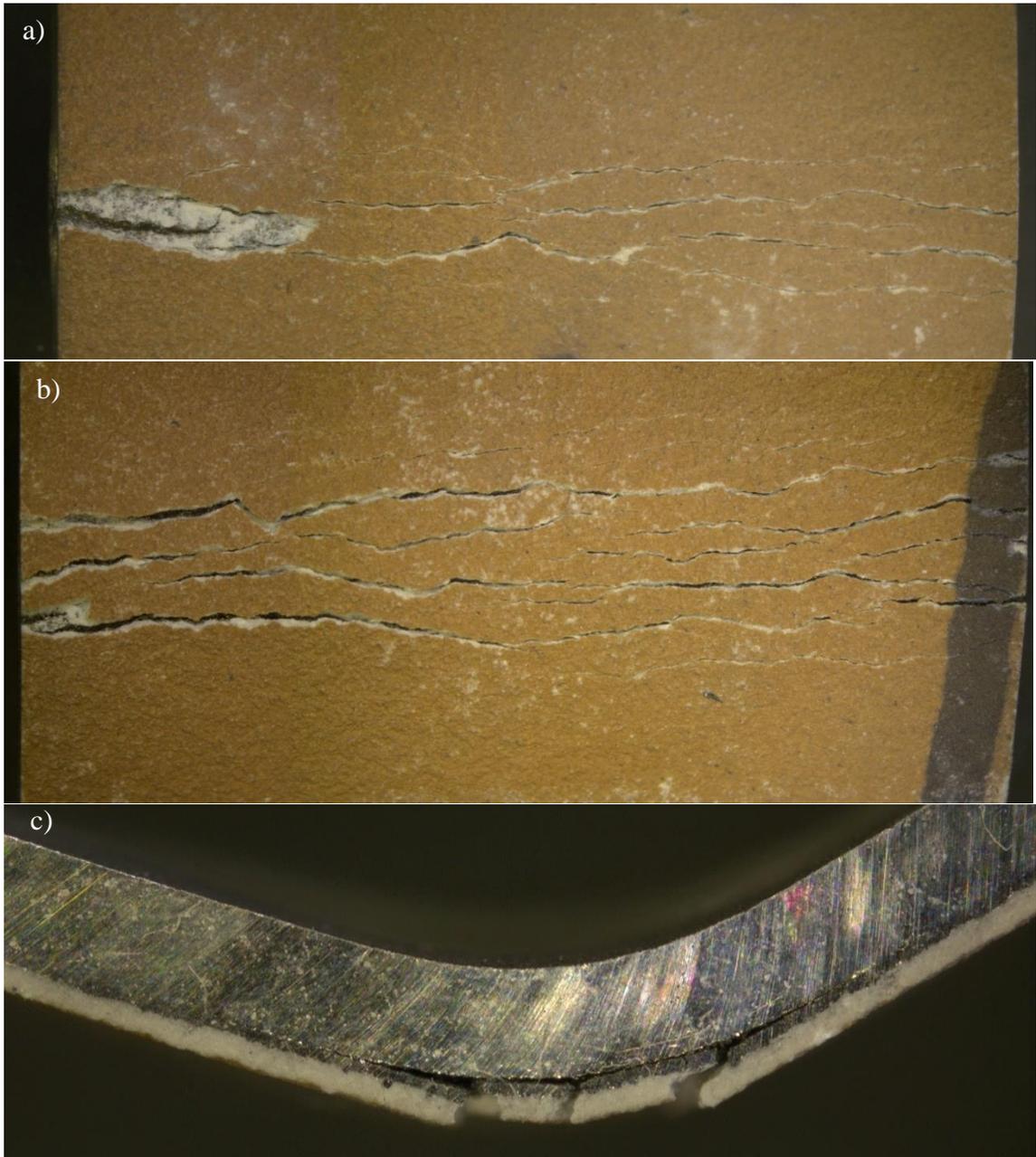


Fig. 17. Sample 2 and sample 3 are example that results of testing can be little different.

On sample 2 on the figure 17, we can see that part of the coating peeled off from the substrate while cracks were creating during the bend test. The samples which are more rusted reveal contamination from exhaust gases. This provides information about utilization and means that such sample was used for many flight hours before testing.

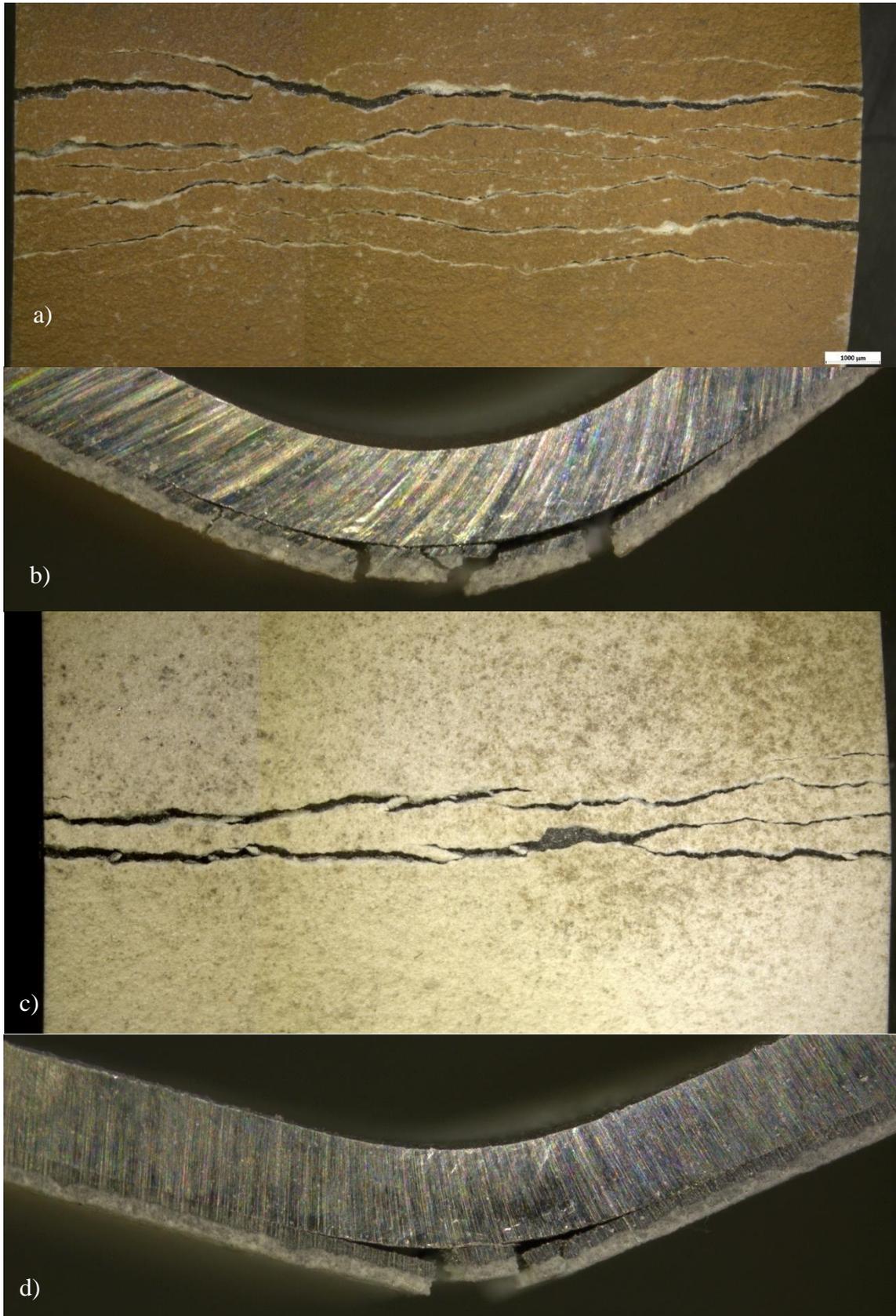


Fig. 18. The Comparison of sample 4 from used combustion chamber (top picture) and sample from new unused component from aircraft. We can see that new sample much lower density of cracks on same area size. There are just 2 main big cracks while on old sample we can see several cracks near main big crack.

5.2. Data analysis

Mechanical characteristic of materials are strength, flexibility, toughness and plasticity but there are more characteristics which provide important information and are in material sheets. These depend on preparation and shape of samples, conditions etc. The bend test is standardized testing method and it belongs to the short term testing method with dynamic nature of the applied force which is variable with time.

As can be seen in previous chapter, samples were tested in equipment ZUZ 200. The length of sample is L . The span, which is distance between the support rollers, is 14 mm. And the test is carried out by a process of controlled deformation. The loading of sample, which can be labeled as beam, must be at constant speed, in this case it is 0.0166 mm/s. The beam is subjected to a controlled load F . The deflection in the middle of sample increases evenly over the time of loading.

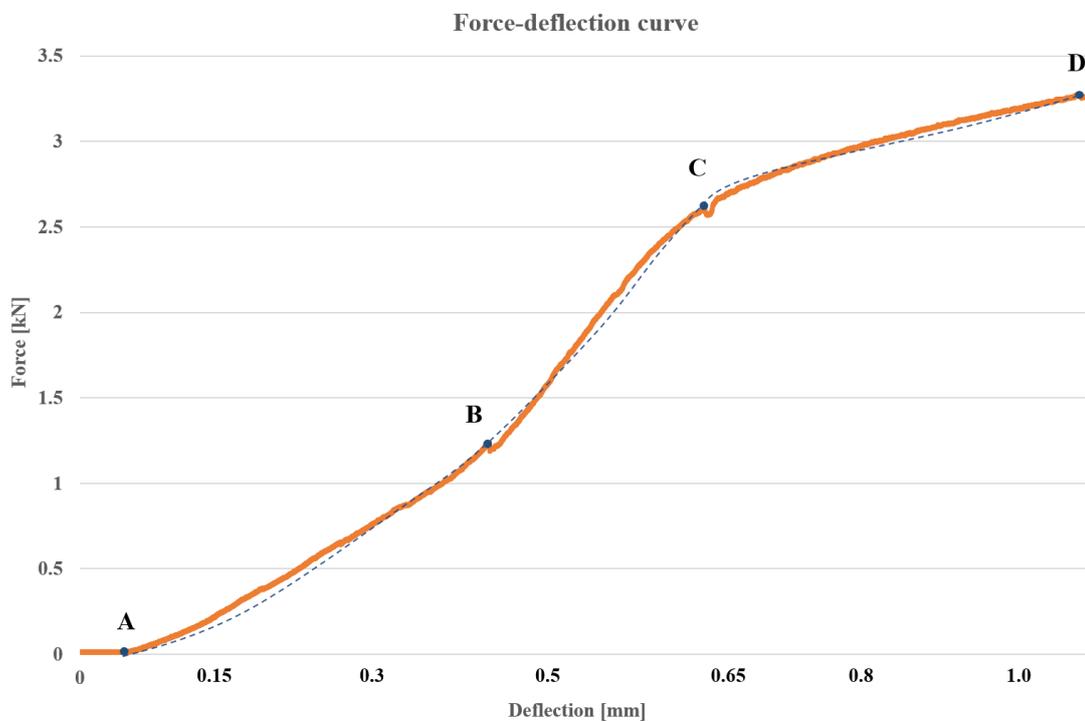


Fig. 19. The graph of the force-deflection curve. During the test, three main characteristics were recorded – time, deflection and force. Just two of them were used to create graph.

While assessing the graph in the figure 19, we can carry forward two of the main characteristics which were recorded to see the dependence of the loading force on the deflection. A curve is not linear. We can assume that sample doesn't have constant modulus of elasticity. Its value is declining with growing loading force. At points B and C, we can assume that a material failure has occurred, and the initiation of cracks has started with several fiber fracture events.

The load-displacement relationship in figure 18 is quite revealing in several ways. The closer investigation of the first stage of the curve starting at point A and ending at point B shows that the sample was deformed linearly to accumulate strain energy which causes initiation of cracks. Closer inspection of the beginning of the second stage around point B shows a sharp decrease of load. At the interface between coating and substrate occurs delamination and accumulating strain energy release cause rapid development of crack. In the next stage between point B and C, the value of the load increases which makes the crack to propagate steadily.

From the closer inspection of the graph above we can count flexural strength R_{max} .

$$R_{max} = \frac{M_{max}}{W_o} [MPa]$$

M_{max} is the maximum bending moment and in the three-point bend test we can count it as

$$M_{max} = \frac{F_{max}}{2} \cdot \frac{L}{2} [N \cdot mm]$$

F_{max} is the maximum loading force and W_o is the section modulus. Samples with the shape of a quadrilateral prism with the thickness h and the width b have the section modulus determined as

$$W_o = \frac{b \cdot h^2}{6} [mm^3]$$

In conclusion, we can calculate the flexural strength of the sample as

$$R_{max} = \frac{F_{max} \cdot L}{4} \cdot \frac{6}{b \cdot h^2} \left[\frac{N \cdot mm}{mm^3} \right] = \frac{F_{max} \cdot L \cdot 3}{b \cdot h^2 \cdot 2} \left[\frac{N}{mm^2} = MPa \right]$$

The deflection v_{max} at the centre of the sample is defined as

$$v_{max} = \frac{F \cdot L^3}{48 \cdot E \cdot I}$$

where E is Young's modulus of a material and I is the moment of inertia defined as

$$I = \frac{h^3 \cdot b}{12}$$

We know Young's modulus for Nimonic, but we don't know the exact value of Young's modulus E for such sample. Its value is affected by amount of coating material meaning thickness of bond coat and top coat, but also by process of spraying, how fast droplets fall on substrate and under what size of angle they fall. In conclusion, for every sample Young's module can have little different value. Now we can calculate value of E with equation

$$(y_2 - y_1) = \frac{(F_2 - F_1) \cdot L^3}{48 \cdot E \cdot I} [mm]$$

where F_1 and F_2 are loading forces measured and recorded in graph, while F_2 has bigger value and y_1, y_2 are deflections of sample in different times which occurred when forces

F_1 and F_2 affected the sample. In this range we can consider deformation as elastic and calculate necessary value of Young's module E for one specimen. [30]

At this moment, we can calculate mentioned equations with values from the experimental part for one example. For the calculation of the maximum bending moment we use the maximum controlled loading force $F_{max} = 3.3 \text{ kN} = 3\,300 \text{ N}$ and the length $L = 14 \text{ mm}$, which is the distance between the rollers in the testing equipment ZUZ 200 on which the sample is placed.

$$M_{max} = \frac{F_{max}}{2} \cdot \frac{L}{2} = \frac{3300}{2} \cdot \frac{14}{2} [N \cdot mm] = 11\,550 \text{ N} \cdot mm$$

W_0 , as section modulus, is calculated with values of the thickness of the sample $h = 1,8 \text{ mm}$ and the width of the sample which is $b = 24,5 \text{ mm}$.

$$W_0 = \frac{b \cdot h^2}{6} = \frac{24,5 \cdot 1,8^2}{6} [mm^3] = 13,23 \text{ mm}^3$$

The maximum flexural strength of the sample is calculated as

$$R_{max} = \frac{M_{max}}{W_0} = \frac{F_{max} \cdot L}{4} \cdot \frac{6}{b \cdot h^2} = \frac{3300 \cdot 14 \cdot 3}{24,5 \cdot 1,8^2 \cdot 2} \left[\frac{N}{mm^2} = MPa \right] = 873 \text{ MPa}$$

The sample reached this value at the end of the bend test when coating had cracks and microcracks and eventually for the old specimen, the coating was partly peeled off. This means that the new specimen is able to withstand testing with bending strength 873 MPa without any peel off effect while the old sample has already exceeded its strength options at the same moment.

When we take a look back to the graph at the figure 19, we can see that in the points B and C have occurred major influence on the coating during the test. To find out at which moment the delamination with peel off effect started during testing of the old sample, we would need to evaluate values of the deflection and the loading force in these points on the curve. Further data collection and evaluation would be required to determine how the deflection and the delamination affects the coating in points B and C of the graph. At this moment, the reader should bear in mind that evaluation at such a detailed level is beyond the scope of the study to examine peel off effect.

Now, we are moving on to comparative analysis of the appearance of the samples. To assess detailed photos of coating, the microscope in GE Aviation Czech laboratory was used. Since the photos show very similar results in old samples and different results in the comparison to the new sample, I decided to talk about results obtained from old samples as one group to which I will do comparison to other group where is new sample.

The first set of analyses examines the appearance of old samples before providing the bend test. Four old samples went through similar way of wear. The samples are rusted which provide information about the use of component. The rust says the sample is contaminated from exhaust gases. The more rusted the sample is, the more flight hours it was utilized in the aircraft. If we now turn to the new sample, there is no signs of the rust meaning the coating came out clean from the primary production.

Turning now to evaluate the appearance of samples after the bend test and the reasons why some results are satisfactory and others not. Considering GE Aviation Czech criteria and standards, peeling off of the coating after providing the bend test is unacceptable result and the acceptable one is coating staying attached to the substrate while having a few cracks and microcracks. From the photos of samples, it can be seen that the set of old samples after 3000 flight hours shows signs of the peel off effect. Cracks and microcracks propagated on the coating during the bend test and ended with part of coating peeled off. Referring to GE Aviation Czech criteria, it is unacceptable to have sample with peeled off coating from the substrate. A comparison of the results from new sample presents evidence of accepted result in association GE Aviation Czech standards. The new sample is having lower density of cracks and the coating did not reach peel off effect.

The final part of the comparative analysis needs to consider reasons why some results are acceptable and others not, what impact do these results have on general inspections and the service of the aircraft. Since the old samples showed signs of the peel off effect, we need to find out why such a result came out. We demand to take in the consideration that a coating life of the old samples could be after 3000 flight hours exhausted and increased susceptibility of the delamination and the peel off effect occurs. It is a natural process. After many flight hours, cohesion and adhesion of the coating to the substrate could be reduced. The performed test is condition which new samples need to pass to be able to put into the operation. The results indicate the correct primary production and that its set parameters are acceptable. In group of old samples, it is predicted that results will come out unsatisfactory. But it does not mean the engine will be decommissioned.

5.3. Discussion

The main goal of the current study was to investigate failure mode of atmospheric plasma sprayed coatings from yttrium stabilized zirconia oxide which occur during primary production and in use in applications. After investigating several types of thermal barrier coating techniques, research has shown that use of atmospheric plasma spaying is the best option for utilization in aircraft components, mainly combustion chamber of engine. As mechanical testing method, we select three-point bend test for realization after which we analysed measured data to assess material structure and mechanical behaviour of samples.

Overall, tests of samples have showed as similar measured values and recorded characteristics as graph in figure 19 above. No great difference was observed. The most interesting aspect of the graph is change of quietly stable curve in points B and C which present occurrence of cracks in coating as mentioned previously. The biggest differences in results of the bend test is in material structure of the coating. Every sample has different size of cracks and different crack density on the same size of the area.

An initial objective of the project was to identify if results of bend test have achieved acceptable or unacceptable outcome referring to GE Aviation Czech criteria and standards. It is interesting to note that in all four old samples tested in this study the coating peeled off while cracks and microcracks were propagated. Such results are unacceptable. But we cannot consider these findings as disappointing since old samples were acquired from the component after the first general inspection which takes place after 3000 flight hours. It was predicted that such coating contaminated from exhaust

gases covered by the rust has more problems to withstand the bend test without the peel off effect. While the result from the new sample may be explained by the fact that the sample was obtained after the primary production of the component where the environment is adapted to prevented contamination of coatings.

This study confirms that the failure of plasma sprayed samples can result from the delamination cracking while in the utilization in aerospace applications with difficulties caused by the contamination from exhaust gases. High compressive stresses in the plane cause considerable tensile stresses out of the plane, produce cracking near the rough bond coat's peaks and cracking parallel to the interface. The discrepancy in results of old used samples in comparison to GE Aviation Czech standards may seem as problematic situation, however, it does not make the aircraft engine with the older coating decommissioned. Differences between results in old samples and new sample may have influence future setting of parameters in primary production to prevent possible delamination of the coating after the first general inspection. This is an important issue since there are still some unanswered questions about coating's wear after utilisation in the combustion chamber of the aircraft engine. Additional investigations will be needed to develop a full picture of characteristics of coatings after some time in utilization and how much they are affected by flue gases. In future studies, it might be already possible to utilize a different testing method to find out mechanical properties of coatings.

5.4. Conclusion

- The results of this thesis indicate that the three-point bend test was successfully carried out to show us differences in delamination of old used samples after 3000 flight hours and new sample acquired after primary production.
- The most obvious finding to emerge from thesis is that old used samples are affected by the amount of flue gases contaminated in coating, coating life after hundreds or thousands of hours of use could be exhausted, and susceptibility of the delamination increased while there was an increased peel off effect.
- The premature failure of atmosphere sprayed thermal barrier coating was not induced in the new unused sample from which we can draw following conclusion indicating the primary production of components with the coatings have the set parameters satisfactory.

5.5. Suggestions and Recommendations

This thesis has showed that many questions are in need of further investigation and studies to provide more information about mechanical characteristics of utilized coatings. These findings could suggest how to set parameters in primary production to prevent failure modes. Despite the promising findings and results, further research should be undertaken to investigate how to prevent possible delamination after few hundreds flight hours of aircraft.

Sources

- [1] HEIMANN, Robert B. *Plasma-Spray Coatings: Principles and Applications* [online]. 1996. Federal Republic of Germany: VCH, 1996, s. 13-15 [cit. 2019-12-07]. ISBN 3-527-29430-9. Dostupné z: <https://books.google.cz/books?id=2p6q2HQybs4C&printsec=frontcover&hl=cs#v=onepage&q&f=false>
- [2] FST, Flame Spray Technologies. *Thermal Spray Technology* [online]. France, 2014, 15 [cit. 2019-10-20]. Dostupné z: <https://www.fst.nl/about/thermal-spray-process-what-is-thermal-spray/>
- [3] FAUCHAIS, P. a A. VARDELLE. *Thermal Spray Coatings* [online]. France, 2007 [cit. 2019-11-21]. Dostupné z: https://www.researchgate.net/publication/229592283_Thermal_Spray_Coatings. University of Limoges.
- [4] PAWLOWSKI, Lech. Thermal Spraying Techniques. *The Science and Engineering of Thermal Spray Coatings*. Second Edition. Chichester, West Sussex: John Wiley & Sons, 2008, s. 67-107. ISBN 978-0-471-49049-4.
- [5] FAUCHAIS, P. a M. VARDELLE. *Plasma spraying: present and future* [online]. Great Britain, 1994 [cit. 2019-11-22]. Dostupné z: <http://citeseerx.ist.psu.edu/viewdoc/download?doi=10.1.1.600.9922&rep=rep1&type=pdf>. University of Limoges.
- [6] NOSEK, Martin. *Perspektivní materiály pro technologii HVOF s aplikací v dopravní technice* [online]. Praha, 2016 [cit. 2019-11-07]. Dostupné z: https://dspace.cvut.cz/bitstream/handle/10467/65813/F2-BP-2016-Nosek-Martin-BP_Perspektivni_materialy_pro_tehnologii_HVOF_s_asplikaci_v_dopravni_tehnice_Nosek_2016.pdf?sequence=-1. Bakalářská práce. České vysoké učení technické v Praze. Vedoucí práce Prof. Dr. Ing. Libor Beneš, IWE.
- [7] ČELKO, Ladislav. Technologie žárových nástřiků. *Engineering.sk* [online]. [cit. 2019-11-22]. Dostupné z: <https://www.engineering.sk/clanky2/stroje-a-technologie/3864-technologie-zarovych-nastriku>
- [8] XU, J. L. a K. A. KHOR. Plasma spraying for thermal barrier coatings: processes and applications. XU, Huibin a Hongbo GUO. *Thermal Barrier Coatings* [online]. 2011. Cornwall, UK: Woodhead Publishing, 2011, s. 99-103 [cit. 2019-12-08]. ISBN 978-0-85709-082-9. Dostupné z: <https://books.google.cz/books?id=hEpWAgAAQBAJ&printsec=frontcover&hl=cs#v=onepage&q&f=false>
- [9] HACKETT, C. M. a G. S. SETTLES. The High-Velocity Oxy-Fuel (HVOF) thermal spray - Materials processing from a gas dynamics perspective. *Fluid Dynamics Conference* [online]. 1995. Pennsylvania, s. 1-6 [cit. 2019-11-3]. Dostupné z: https://www.researchgate.net/publication/269056784_The_High-Velocity_Oxy-Fuel_HVOF_thermal_spray_-_Materials_processing_from_a_gas_dynamics_perspective

- [10] HVOF spraying. *Griekspoor thermal coatings* [online]. [cit. 2019-11-17]. Dostupné z: <https://griekspoorthermalcoatings.com/hvof-spraying>
- [11] ŁATKA, L. Thermal Barrier Coatings Manufactured by Suspension Plasma Spraying - A Review. *Advances in Materials Science: The Journal of Gdansk University of Technology* [online]. Warsaw, Poland: De Gruyter Poland, 2018, 29.10.2018, **18**(3), 95–117 [cit. 2019-12-08]. ISSN 2083-4799. Dostupné z: [https://content.sciendo.com/configurable/contentpage/journals\\$002fadms\\$002f18\\$002f3\\$002farticle-p95.xml](https://content.sciendo.com/configurable/contentpage/journals$002fadms$002f18$002f3$002farticle-p95.xml)
- [12] ZHU, Dongming a Robert A. MILLER. *Mechanical Properties of Plasma-Sprayed ZrO₂-8 wt% Y₂O₃ Thermal Barrier Coatings* [online]. Ohio Aerospace Institute, Brook Park, Ohio, 2004 [cit. 2019-12-08]. Dostupné z: <https://ntrs.nasa.gov/archive/nasa/casi.ntrs.nasa.gov/20040191421.pdf>. U.S. Army Research Laboratory. Glenn Research Center, National Aeronautics and Space Administration.
- [13] GRISAFFE, Salvatore J. a William A. SPITZIG. *Preliminary investigation particle substrate bonding of plasma-sprayed materials: NASA TECHNICAL NOTE NASA TN D-1705* [online]. Washington, D.C.: Lewis Research Center, Cleveland, Ohio, 1963 [cit. 2019-12-10]. Dostupné z: <https://books.google.cz/books?id=67PxmHrEDNgC&printsec=frontcover&hl=cs#v=onepage&q&f=false>
- [14] PLANQUES, Pierre, Vanessa VIDAL a Philippe LOURS. Characterization of the mechanical properties of thermal barrier coatings by 3 points bending tests and modified small punch tests. *Surface & Coatings Technology* [online]. 2017, 25. 12. 2017, (332), 40-46 [cit. 2020-01-06]. ISSN 0257-8972. Dostupné z: <https://www.sciencedirect.com/journal/surface-and-coatings-technology>
- [15] QIAN, G., T. NAKAMURA a C. C. BERNDT. Tensile Toughness Test and High Temperature Fracture Analysis of Thermal Barrier Coatings. *Acta Metallurgica Inc* [online]. Great Britain: Elsevier Science, 1997, 11. 5. 1996, 45(4) [cit. 2020-01-06].
- [16] BERNDT, Christopher C. Instrumented Tensile Adhesion Tests on Plasma Sprayed Thermal Barrier Coatings. *J. Materials Engineering* [online]. New York: Springer-Verlag New York, 1989, 11(4), 275-282 [cit. 2020-01-06]. Dostupné z: <https://link.springer.com/content/pdf/10.1007/BF02834137.pdf>
- [17] CAO, X. Q., R. VASSEN a D. STOEVEER. Ceramic materials for thermal barrier coatings. *Journal of the European Ceramic Society* [online]. 2004, (24) [cit. 2020-01-06]. DOI: 10.1016/S0955-2219(03)00129-8.
- [18] AZADI, M., G. H. FARRAHI a A. MORIDI. Optimization of Air Plasma Sprayed Thermal Barrier Coating Parameters in Diesel Engine Applications. *Journal of Materials Engineering and Performance* [online]. 2013, 22(11) [cit. 2020-01-06]. DOI: 10.1007/s11665-013-0629-5. Dostupné z: https://www.researchgate.net/publication/247161734_Optimization_of_Air_Plasma_Sprayed_Thermal_Barrier_Coating_Parameters_in_Diesel_Engine_Applications

- [19] BOUZAKIS, K. D., A. LONTOS a N. MICHAILEDIS. Determination of mechanical properties of electron beam-physical vapor deposition-thermal barrier coatings (EB-PVD-TBCs) by means of nanoindentation and impact testing. *Surface and Coatings Technology* [online]. 2003, 75-80 [cit. 2020-01-06].
- [20] DAVIS, J. R. a Thermal Spray Society Training Committee. *Handbook of Thermal Spray Technology*. Third printing. United States of America: ASM International, 2009.
- [21] ELSHALAKANY, Abou Bakr a T.A. OSMAN. Comparative study between high-velocity oxygen fuel and flame spraying using MCrAlY coats on a 304 stainless steel substrate. *The Journal of Materials Research and Technology* [online]. Brazil, 2019, September 2019, 8(5) [cit. 2020-01-08]. DOI: 10.1016/j.jmrt.2019.07.035. ISSN 2238-7854. Dostupné z: <http://www.jmrt.com.br/en-comparative-study-between-high-velocity-oxygen-articulo-S2238785419306180>
- [22] ZHUA, Qi, Wei HE a Jianguo ZHU. Investigation on interfacial fracture toughness of plasma-sprayed TBCs using a three-point bending method. *Surface and Coatings Technology* [online]. 2018, 15. 11. 2018, (353), 75-83 [cit. 2020-04-08]. ISSN 0257-8972. Dostupné z: <https://www.sciencedirect.com/science/article/pii/S0257897218308958>
- [23] R. A., Miller. Thermal barrier coatings for aircraft engines: history and directions. *Journal of Thermal Spray Technology* [online]. 1997, March 1997, 35(6), 35-42 [cit. 2020-04-08]. DOI: <https://doi.org/10.1007/BF02646310>. ISSN 1059-9630. Dostupné z: <https://link.springer.com/article/10.1007/BF02646310>
- [24] LASHMI, P. G. a P. V. ANANTHAPADMANABHAN. Present status and future prospects of plasma sprayed multilayered thermal barrier coating systems. *Journal of the European Ceramic Society* [online]. 2020, July 2020, 40(8), 2731-2745 [cit. 2020-10-13]. ISSN 0955-2219. Dostupné z: [doi:https://doi.org/10.1016/j.jeurceramsoc.2020.03.016](https://doi.org/10.1016/j.jeurceramsoc.2020.03.016)
- [25] JOHNSON, W. Steven. *Delamination and Debonding of Materials: A Symposium Sponsored by ASTM Committees D-30 on High Modulus Fibers and Their Composites and E-24 on Fracture Testing*. Pittsburg: American Society for Testing and Materials, 1985. ISBN 0-8031-0414-6.
- [26] WANG, Bo a Thomas SIEGMUND. A modified 4-point bend delamination test. *Microelectronic Engineering (MICROELECTRON ENG)* [online]. 2008, 37(85), 477-485 [cit. 2020-10-29]. ISSN 0167-9317. Dostupné z: [doi:10.1016/j.mee.2007.08.010](https://doi.org/10.1016/j.mee.2007.08.010)
- [27] WANG, Luyi. Failure Behavior of Plasma-Sprayed Yttria-Stabilized Zirconia Thermal Barrier Coatings Under Three-Point Bending Test via Acoustic Emission Technique. *Journal of Thermal Spray Technology: J THERM SPRAY TECHN* [online]. 2017, 13.12.2016, 26(1) [cit. 2020-11-2]. ISSN 1059-9630. Dostupné z: [doi:10.1007/s11666-016-0497-2](https://doi.org/10.1007/s11666-016-0497-2)
- [28] EVANS, A. G. a J. W. HUTCHINSON. The mechanics of coating delamination in thermal gradients. *Surface and Coatings Technology* [online]. 2007, 25. 6. 2007, 18(201), 7905-7916 [cit. 2020-11-11]. ISSN 0257-8972. Dostupné z: [doi:https://doi.org/10.1016/j.surfcoat.2007.03.029](https://doi.org/10.1016/j.surfcoat.2007.03.029)

- [29] BRENNEN, Christopher Earls. CAVITATION AND BUBBLE DYNAMICS [online]. Oxford University Press, 1995 [cit. 2020-11-23]. ISBN 0-19-509409-3. Dostupné z: <https://authors.library.caltech.edu/25017/1/cavbubdynam.pdf>
- [30] SOBOTOVÁ, Jana. Nauka o materiálech I. a II.: Cvičení. Praha: Česká technika, 2016. ISBN 978-80-01-05550-2.
- [31] NIMONIC® alloy 80A [online]. In: . Special Metals Corporation, 2004, s. 1-24 [cit. 2020-11-23]. Dostupné z: <https://www.specialmetals.com/assets/smc/documents/alloys/nimonic/nimonic-alloy-80a.pdf>
- [32] ŘEZNÍČEK, Jan. Pružnost a pevnost I.: Přednášky. 4. doplněná verze. Praha: České vysoké učení technické v Praze, Fakulta strojní, 2013.