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STUDENT RESEARCH THESIS

Tribological investigation of tool steel with advanced superhard coatings

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CONTENT

1. Introduction	5
2. Literature review	6
2.1. The characteristic operating conditions and the most common causes of damage and failure of cold work tool steels	6
2.1.1. Coatings failures that occurs during the working process of the coated surface	6
2.2. Coating processes.....	9
2.2.1. Surface coating.....	10
2.2.2. Coating technologies	10
2.3. Ceramic hard and superhard coatings for improved wear resistance.....	13
2.4. Material testing methods used for characterizing the coating performance	14
2.4.1. Microscopic investigation	14
2.4.2. Ball-on-disc test	15
2.4.3. Calotest.....	16
2.4.4. Scratch test	17
3. Experimental work	18
3.1. Investigated material	18
3.1.1. Sample processing.....	19
3.1.2. Heat treatment	21
3.1.3. Polishing.....	22
3.1.4. Coating process	23
3.1.5. Microstructure of the coated samples	26
3.1.6. Surface preparations.....	27
4. Test results and evaluation	29
4.1. Hardness test	29
4.2. Calotest (ball cratering test)	30
4.3. Scratch test	33
4.3.1. Increasing loading scratch test	34
4.3.2. Constant load scratch test.....	38
4.4. Ball-on-disc test	44
5. General review and evaluation of the integrated test results	50
6. Summary	51
7. Application potential of the results of the research work.....	52
8. Further direction of research work	52
Acknowledgement.....	54
References	55

1. INTRODUCTION

Nowadays, different Advanced High-Strength Steel (AHSS) are utilizing everywhere, also the strength of structural parts made of AHSS is continuously increasing. In crash-relevant automotive structural parts, AHSS and UHSS (ultra-high strength steels) are known as standard sheet material.

To maintain high quality and efficiency for these challenging applications, the tools that are used to manufacture such components, need even stronger surface solutions. When such kinds of components processed, the high strength of metal causes significant stresses on the tool surface, especially in highly loaded zones which can result in rendered fatigue, wear, corrosion, scratching, and other issues.

The primary goal of coating a tool is to improve wear resistance, reduce adhesion risk, and decrease sticking between the tool and the workpiece. The result of using coated tools is often a combination of increased productivity and a higher quality of processed components.

PVD coatings are a productive and revolutionary technology for decreasing friction and preventing the substrate surface from wear, corrosion, and other problems. Wear can be defined under frictional conditions and it results in material loss, material surface deterioration, machining tool lifetime reduction, and increased failure risk. There have been recent developments and growth in this area, and several coating methods and new deposition techniques, such as PVD, CVD, and PACVD (plasma assisted chemical vapour deposition) are increasingly applied in the industry.

Combined processes have a high potential for the future treatment of metals because, in an increasing number of cases, the resistance of metals against complex and cyclic loads is no longer sufficient. Duplex technology, especially in the automotive industry is represented mostly by the combined processes of a PVD or PACVD coating and a previous plasma nitriding, aiming at enhancing the mechanical, chemical and physical performance of the surface layer in a new way. These technologies alone have their characteristic own shortcomings – e.g. during nitriding lower maximum hardness, and wear resistance can be achieved, while applying PVD processes alone the hard ceramic coatings deposited on a ductile metallic substrate may lead to spalling, debonding, or weak adhesion between the coating/substrate interface. Duplex treatments, combining these technologies, enables reducing their individual limits and building surfaces of enhanced tribological and mechanical properties.

The objective of our research work is to compare the tribological behaviour of two industrially used advanced duplex coatings combining the advantage of nitriding and ceramic coating. The comparison is practically realized by characterizing their tribological performance by wear- and scratch tests, in addition by some auxiliary tests like Calotest, hardness test, and microscopic investigations, etc.

The executed work proved a revealed improvement of wear resistance of the tool steel by applying these advanced ceramic coatings and gives useful information on the tribological performance of these coating, which provides the possibility for further improvement of the current industrial coating technology.

2. LITERATURE REVIEW

2.1. The characteristic operating conditions and the most common causes of damage and failure of cold work tool steels

Each type of tool steel is designed to perform well under the various operating conditions. Even when they are produced to the highest standards, each type of steel has its operational limits, and factors such as wear under recommended operating conditions are unavoidable. During manufacturing or application, problems may arise that can minimize the lifespan of tool or even result to fail completely [1]. The list of problems are:

- Fatigue
- Wear
- Corrosion
- Scratching
- Other issues

2.1.1. Coatings failures that occurs during the working process of the coated surface

Abrasive wear

Abrasive wear is defined as hard particles or hard protuberances that are forced against and move along a solid surface, causing damage and progressive material loss to the solid surface. In tooling, abrasive wear is often found when a nonmetallic particle, such as an oxide or ceramic grain, scratches the surface of the tool. These hard particles scour the tool surface as shown schematically in Fig. 1. This is due to the presence of impurities such as dust or oxidation in metal-forming operations, or

by the tooling operation itself when ceramic or other nonmetallic materials are shaped in a tool-steel die [2].

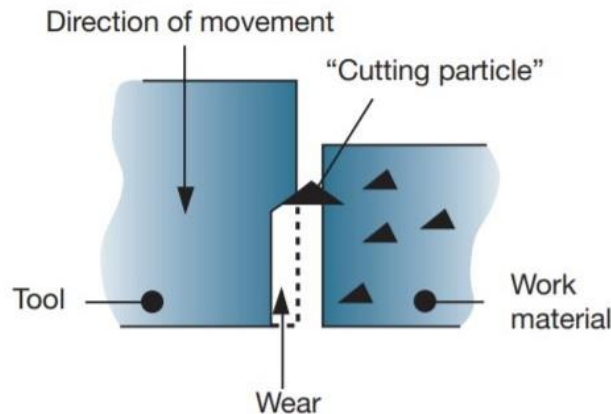


Fig. 1. Schematic representation of abrasive wear [3]

Adhesive wear and Galling

Adhesive wear or also known as galling (Fig. 2.) is the transition of material from one metallic surface to another as they slip over each other. It is caused by the micro welding process (solid-phase welding).

Metallic surfaces are never fully smooth; there are microscopic asperities on them. At these asperities, bonding can arise between the two materials, which may be stronger than the basic strength of the weaker material in contact. If these two surfaces move relative to each other, the weaker of the two materials in contact will fail, and the material will be transferred to the other. Tiny pieces of tool steel are torn from the tool surface if the microweld cracks on the tool side, causing adhesive wear. When the microwelds on the production material side fail, production material fragments can gall onto the tool surface.

Chipping may also be caused by adhesive wear. In the early stages, the adhesive wear is dominant, but a fatigue mechanism eventually takes over. Microcracks begin to form, and as they do, they deepen and spread. Large-scale spalling (chipping) or even catastrophic failure may result from the cracks. Soft, adhesive metallic work materials like aluminum, copper, stainless steel and low carbon steel are commonly associated with adhesive wear and galling.

Galling, on the other hand, is an issue that occurs during the manufacturing of components made of high-strength production materials like advanced high strength steel [2] .



Fig. 2. Galling [3]

Chipping

Chipping (Fig. 3) is a common occurrence after a tool has been in use for a short period of time. Low cycle fatigue is the cause of this failure. Microcracks begin on the tool's active surface, spread, and eventually result in pieces chipping out along edges and corners. It is critical to make microcrack initiation and propagation more difficult in order to achieve good chipping resistance. High ductility is one of the tool steel properties that helps it avoid chipping [2].

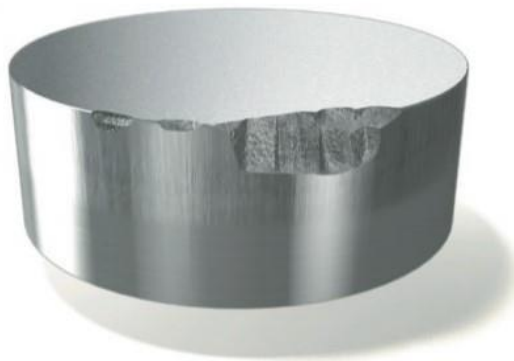


Fig. 3. Chipping [4]

Cracking

Cracking (Fig. 4) is a form of failure that occurs on its own and typically necessitates the replacement of the instrument. The mechanism that causes this form of failure is unstable crack propagation. The presence of stress concentrators, such as grinding marks and machining marks, or design features such as sharp corners or radii, greatly aids the formation of cracks. EDM layers on the tool surface are another common source of damage. It's worth noting that the low hardness would have a negative impact on the resistance to other failure mechanisms. As a result, working with low hardness is usually not a good idea. It's preferable to use steel that has high microstructural durability [2].



Fig. 4. Cracking [3]

The most critical matter in modern mechanical engineering is to improve wear resistance. Improving the wear resistance (WR) of machine steels is a key factor in boosting the efficiency and competitiveness of advanced manufacturing processes. Surface treatments, especially nitriding, are frequently used to help increased surface hardness and thus increase tool wear life [3].

Coatings or changes to the metallurgy or chemistry of the substrates may help to prevent wear in these tribosystems, extending the efficiency and lifetime of tool steels.

2.2. Coating processes

Surface engineering encompasses surface modification, thin-film deposition, and coating deposition. These technologies can increase the wear and corrosion resistance of engineering components, extending product life and protecting materials.

Surface coatings and surface modifications are the two primary surface engineering methods for improving the surface properties of bulk materials [5].

Surface engineering may produce practical films or coatings that have improved properties including high-temperature resistance, corrosion resistance, anti-wear, and fatigue resistance.

Films or coatings have superior properties and a much thinner thickness than bulk substrates, but they can take on most of the surface loadings of workpieces [6].

The classification of the surface engineering technologies is shown in Fig. 5, where the yellow colour indicates the technological processes applied during producing the ceramic coatings investigated in the current research work.

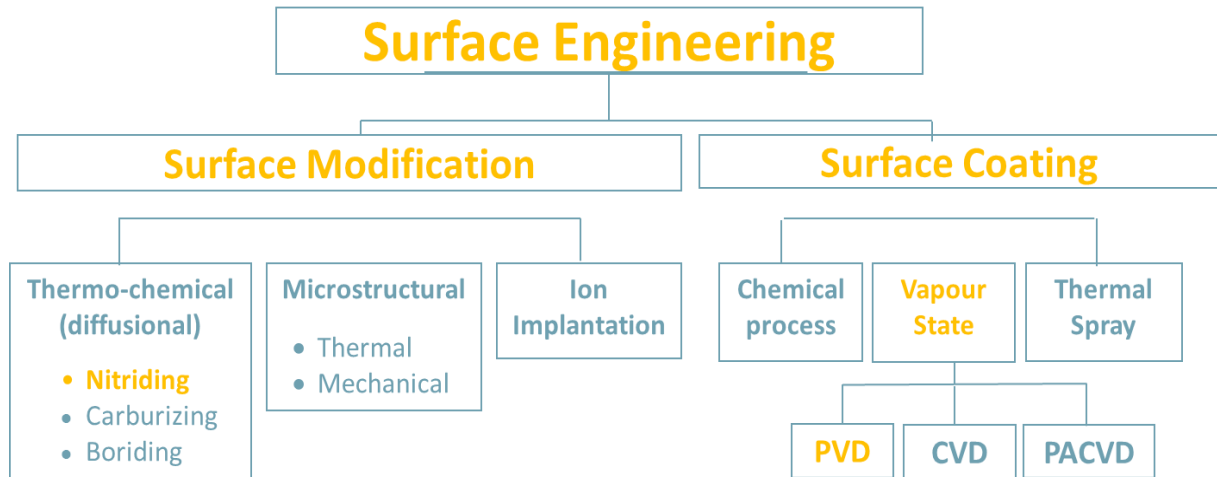


Fig. 5. Classification of surface engineering technologies [7]

2.2.1. Surface coating

The application of coatings to the surface of engineering parts is one of the most effective ways to shield them from the mentioned types of damage and failure. Coatings are used among the others to protect the substrate by physically isolating it and preventing aggressive organisms from spreading to its surface.

One of the most efficient ways to reduce friction and protect contact surfaces from wear is to use coatings.

In recent decades, several new deposition technologies have been developed, and there are numerous tribological coatings available [8]. Vapor deposition PVD and CVD techniques are used to deposit coatings and thin films in electronics, optics, data storage, and decorative applications, but particularly in tribological applications. The various coatings or coating systems are deposited using very different plasma vapor deposition PVD and chemical vapor deposition CVD processes or process combinations hybrid processes.

These processes, along with the process parameters chosen, allow for a wide range of coating properties and performance, as well as laboratory or application-specific conditions [9].

2.2.2. Coating technologies

Physical Vapor Deposition (PVD)

Since 1980, tool coatings created using physical vapor deposition (PVD) technologies have become a reality, and an industry has grown up around PVD coated tools thanks to the early pioneers' work. In most PVD hard coating

techniques, the metal species are vaporized, and gas is fed into the coating chamber, where it reacts with the metal species to form the desired compound. Four specific types of equipment are currently in use for the deposition of PVD tool coatings, all of which fall within the broad category of ion plating. The vaporization method of the source material, either evaporation or sputtering; the manner in which the plasma is created; and the number and types of ions, electrons, and gas atoms that make up the plasma are the differences between the four classes. Low-voltage electron beam evaporation, cathodic arc deposition, triode high-voltage electron beam evaporation, and balanced and unbalanced magnetron sputtering are the four PVD hard coating techniques. Although there are many similarities between these four PVD methods, there are also many differences that can affect the types of films that can be deposited in these systems. Each approach has benefits and disadvantages, and no single method is suitable for all applications. The process of low-voltage electron beam evaporation is extremely effective at ionizing the evaporating reactive gas atoms, with an average ionization efficiency of 50%. A very reliable method for creating a very hard and smooth Nickel, which has become the industry norm, is low-voltage electron beam deposition. Sputter deposition is one of the most complicated processes and is more costly in many situations. Sputter deposition, on the other hand, allows for better control of multielement film composition and a wider range of materials that can be deposited [10].

The cathodic arc deposition technique is one of the most important PVD techniques, as it is primarily used to prepare strong, wear-resistant coatings for tools and other mechanical parts. There are many benefits, such as high ionization and good homogeneity of coatings on complex structure substrates, but there are also some drawbacks. However, the key downside is the formation of macroparticles, which degrades the consistency of the deposited coatings. Hard coatings such as titanium nitrides and carbides, zirconium, molybdenum, and chromium nitrides and carbides, Al_2O_3 films, and diamond-like carbon films have all been successfully deposited using arc evaporation [10].

Chemical Vapor Deposition

CVD is a technique for altering the surface properties of engineering components by depositing a layer or layers of another metal or alloy at a high temperature by chemical reactions in a gaseous medium surrounding the component. Chemical vapor deposition is a process in which a mixture of gases interacts with the surface of a substrate at a relatively high temperature, resulting in the decomposition of some of the gas mixture's constituents and the creation of a solid film of a metal or a compound on the substrate. A wide variety of materials, including metals, compounds, ceramics, powders, and whiskers, can be deposited using the CVD

technique. We want to mention the typical materials deposited by CVD and applications of the CVD technique in Table 1 [10].

Table 1. Characteristic materials deposited by CVD [10]

Metals	Al, As, Be, Bi, Co, Cr, Cu, Fe, Ge, Hf, Ir, Mo, Nb, Ni, Os, Pb, P, Re, Rh, Ru, Sb, Si, Sn, Ta, Th, Ti, U, V, W, Zr, also carbon and boron
Compounds	II-VI and III-V compounds, borides, carbides, nitrides, and silicides of transition metals, as well as sulfides, phosphides, aluminides, etc.
Ceramics	Al_2O_3 , AlN, B_2O_3 , BN, SiC, Si_3N_4 , UO_2 , Y_2O_3 , ZrO_2 , etc.
Application of CVD techniques	Tribological coatings, wear resistant coatings, high temperature coatings, photovoltaic films

CVD demands relatively high temperatures, with the coating temperature usually ranging from 500 to 1000 °C. This condition limits the types of substrates that can be used for deposition. Because of their high thermal and chemical stability, most ceramic materials, graphite, and refractory metals such as tungsten and molybdenum are found to be very suitable in modern CVD process environments. Steels were also used successfully, but there are still some things that can be done to improve efficiency. For example, most steels other than austenitic and ferritic steels undergo solid-state phase transformation at temperatures between 700 and 800 °C. CVD is most well-known and widely used in the production of coated cemented carbide cutting equipment. These tools are made of tungsten carbide – cobalt alloys with a wear-resistant refractory coating applied by CVD. TiC, TiN, and Al_2O_3 as well as their variations, are widely used coatings [10].

Furthermore, PVD and CVD processes have a high degree of flexibility in terms of coating composition and structure, and these processes are now successfully used to coat a wide variety of mechanical components. The most popular PVD and CVD coating products are nitrides (Nitrides (TiN, CrN, etc.), carbides (TiC, CrC, W_2C , WC/C, etc.), oxides (e.g., alumina), or combinations of these.

Molybdenum disulfide (MoS_2), diamond-like carbon (DLC), and diamond, in addition to these materials, are also very effective [11].

Plasma-Assisted Chemical Vapor Deposition (PACVD). Diamond pre-cursor material had to be condensed as a continuous film onto the surface of the portion to

be coated from a vapor phase in order to use diamond-coat equipment and engineered components. The vapor-deposited material must also condense with the diamond structure and morphology during the deposition process. Diamond is a metastable form of carbon that tends to assume its most thermodynamically stable state or shape — graphite — when condensed by vapor or a flow of energy particles. The use of PACVD techniques to create diamond films is limited to applications where the substratum can be heated to temperatures above 1093 °C. As a result, diamond films deposited using PACVD techniques are suitable for use in semiconductor structures and devices. Continuous diamonds and diamonds on component surfaces, including carbon films, are now possible thanks to recent advancements in plasma-assisted chemical vapor deposition (PACVD) and ion beam enhanced deposition technologies. Their new films have many of the same mechanical, thermal, optical, and electrical properties as single-crystal diamond, enabling diamond facing of precision tools and wear parts, optical lenses and modules, computer disks, and advanced semiconductor devices [10].

2.3. Ceramic hard and superhard coatings for improved wear resistance

Ceramic coatings have been commonly used in a variety of engineering systems, such as shielding structural materials from the elements, extending the life of manufacturing tools by improving wear/corrosion resistance, and improving energy storage and/or conversion performance [12].

Hard coatings are used on more than 40% of cutting tools to minimize running costs, energy consumption, and environmental degradation. Coatings are classified as hard ($H < 40$ GPa), superhard ($40 \leq H \leq 80$ GPa), and ultrahard ($H > 80$ GPa) layers based on their hardness values (H) [13].

Hard coatings are thin films applied on tool substrates to improve desired properties such as hardness, friction, wear resistance, and corrosion resistance without affecting the bulk material's properties. Hot hardness, high adhesion to the ground, and chemical stability are the most significant coating properties from a functional standpoint. Optimal coating thickness, fine microstructure, and residual compressive stresses can all help to improve coating quality. Ceramic materials are usually the only ones that can meet these requirements. In practice, metal nitrides transition, as well as carbides and oxides, are the only options. These materials can be safely deposited as thin films using CVD or PVD techniques.

Unlike CVD processes, the PVD process starts with a solid (e.g. titanium), and the volatile elements are usually introduced as a gas (e.g. nitrogen). Hard PVD coatings have many advantages: low deposition temperature conserves cemented carbide

durability, they can be applied to sharp cutting edges, compressive residual stress prevents microcracks, they have finer grains (smoother surface), higher microhardness, nonequilibrium compositions that are not possible with CVD. Hard PVD coatings have the drawbacks of thickness restriction due to residual stress and, in some situations, poor adhesion. Another disadvantage is that PVD deposition is a line-of-sight process, which means that very complex geometries cannot be uniformly coated [14].

Because of their high strength and mechanical/chemical stability, **superhard** ceramic materials are still in demand in modern technology. Diamond, cubic boron nitride, and high-boron compounds, for example, have a Vickers microhardness of greater than 30 GPa and are intended to be used in a variety of cutting instruments, molds, and protective coatings. Due to their heavy covalent bonding and low diffusivity of their constituents in solids, even at high temperatures, these materials are difficult to shape and sinter. As a result, to prepare bulk bodies or coatings of these materials, special consolidation techniques are needed.

Solid materials containing light elements such as B, C, N, and O are known to have super hardness, and their combination is expected to produce even more super hard and tough materials. Transition metal borides, carbides, and nitrides are also hard materials with microhardnesses of 20-30 GPa. In comparison to other ceramic oxides, these compounds are often difficult to sinter under normal pressure. As a result, for consolidation, hot pressing or hot isostatic pressing is used. Protective hard coatings of the above superhard materials are commonly formed using chemical/physical vapor deposition (CVD/PVD) techniques on metals, cermets, or ceramics [15].

2.4. Material testing methods used for characterizing the coating performance

2.4.1. Microscopic investigation

The purpose of the microscope is to reveal and study objects being smaller than the resolution limit of the human eye (0.1 mm) [16].

The basic principle of the light microscope is shown in Fig. 6. An image of the object (specimen) is formed by the objective lens, which typically provides magnification in the range of $10\times$ to $100\times$. This magnified image is then viewed through the eyepiece (ocular), whose magnification is usually $10\times$ [16].

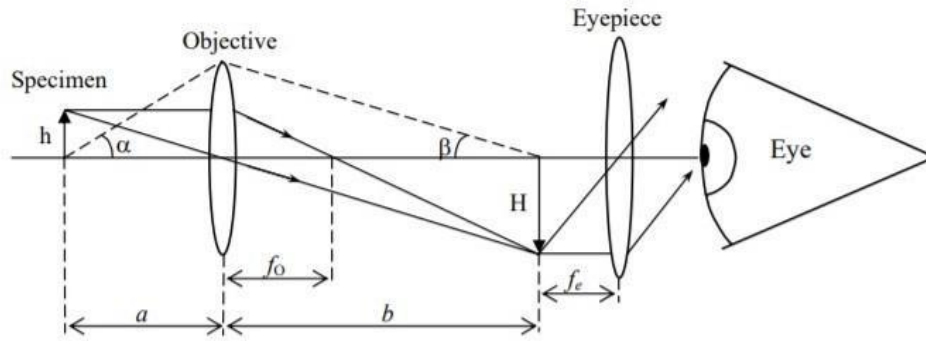


Fig. 6. Principle of the light microscope

The microscope consists of two lens systems, the objective, and the eyepiece. The objective forms a real image of the specimen, and this image is viewed through the eyepiece forming a virtual image at infinity. Typically total magnifications are in the range of $100\times$ to $1000\times$. There is 2 kind of optical microscope:

- Inverted microscope.
- Stereo microscope

The required test equipment

Inverted microscope. Available objective lenses: $25\times$, $50\times$, $100\times$, $200\times$, $500\times$, $1000\times$. Investigation can be accomplished in bright field and dark field modes or using polarized light. Digital screen and image processing software [16].

2.4.2. Ball-on-disc test

The ball-on-disc wear test method is a tribological test that uses a spinning disk to investigate the wear behavior of materials during sliding (Fig. 7.) [17].

Working principle of the ball-on-disc test. To ensure that all dirt and foreign matter are removed from the surface, the specimens are washed and dried immediately before testing. This phase requires the use of non-chlorinated cleaning agents and solvents that do not form films. The disk should then be securely placed in the holding system so that it is fixed perpendicular to the resolution axis. The next step is to secure the pin specimen in its holder and, if possible, change it so that it is perpendicular (1°) to the disk surface when in contact, in order to preserve the required contact conditions. Following that, we'll set some parameters and start the test with the specimens in contact while under load. When the desired number of revolutions is reached, the test is stopped. It is not recommended that the test be stopped or restarted. Finally, the specimens are removed and any loose wear debris is cleaned away.

We can also investigate the wear debris and learn more about the type of wear mechanism, as well [18].

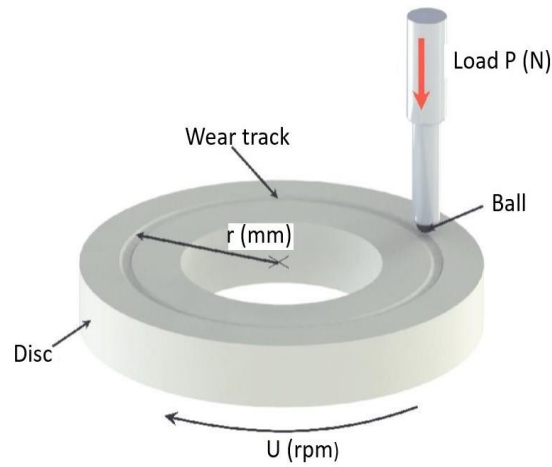


Fig. 7. Principle of the ball-on-disc test [19]

2.4.3. Calotest

Calotesting is a simple and fast method of determining the thickness of mono- and multilayer coatings. The Calotest approach is depicted schematically in Figure 8. It's commonly used to calculate coating thicknesses ranging from 0.1 to 50 μm . The Calotest can be used to evaluate a wide range of coatings produced by Chemical Vapour Deposition (CVD), Physical Vapour Deposition (PVD), and other deposition processes [20].

The coating surface is ground with a wide diameter (approximately 2050 mm) steel or ceramics bearing ball to create a crater that hits the substrate. The introduction of a few drops of a diamond abrasive solution is recommended for the proper grinding effect of hard coating [21].

To calculate the coating thickness, we will need to know the sphere's radius and the outer and inner diameters of the worn groove we've created (the boundary between the coating and substrate). A microscope with a magnification of 100x500x is used to measure the diameters [20].

To look at it another way, the Calotester was designed and built to measure the thickness of thin single and multilayer coatings using abrasive grinding before the substrate is exposed. The coating is involved in sliding friction, for this reason, resulting in abrasive wear caused by a hardened sphere rotating around its base.

As a result, circular (on planar surfaces) or elliptical (on cylindrical surfaces) grooves are obtained [20].

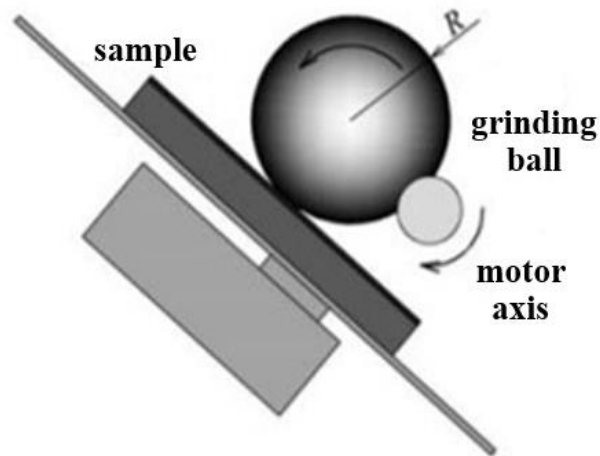


Fig. 8. Schematic principle of ball cratering method (Calotest) [21]

2.4.4. Scratch test

Scratch testing is an effective method for determining a coating's cohesive or adhesive properties. Hard metal or diamond spherical tipped indenter with a tip radius of $200\ \mu\text{m}$ is used to exert a load to the coating surface (Fig. 9.).

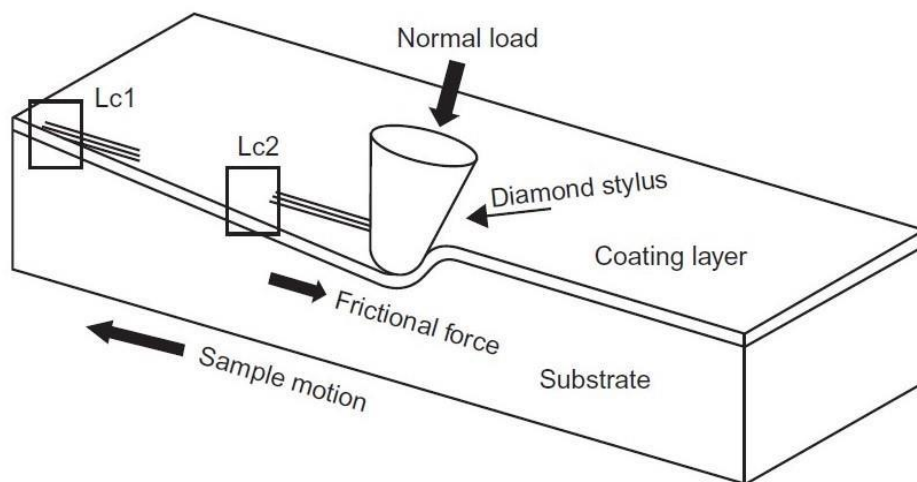


Fig. 9. Working principle of scratch tester [22]

The load may be set constant or gradually increased, depending on the requirement. Hard object, such as an indenter or a stylus, is pushed onto the material surface under normal load and rotated horizontally to create a scratch on the specimen's surface during the test. Scratches appear as a result of various load thresholds, referred to as critical loads.

Optical microscopy, acoustic emissions, or friction force measurements are often used to assess the critical load. The load whereby the first crack appears is known as the "lower critical load," and it is a measure of the coating's cohesion strength. The load at which the coating fully peels away from the substrate surface is known as the "upper critical load," and it is a measure of the coating's adhesion strength to the substrate [23].

3. EXPERIMENTAL WORK

3.1. Investigated material

K110 cold work tool steel was used as the substrate material of the coated specimens. It is a dimensionally stable, ledeburitic 12% chromium content steel with very good wear resistance and acceptable toughness [24]. The standard chemical composition relating to the main alloying elements is given in Table 2.

Table 2. Standard chemical composition in weight [24]

C	Si	Mn	Cr	Mo	V
1.55	0.3	0.3	11.3	0.75	0.75

High-performance cutting tools (dies and punches), die-cutting tools, woodworking tools, shear knives for thin pieces, thread rolling tools, drawing, deep drawing and extrusion press tools, pressing tools for the ceramic and pharmaceutical industries, cold rolling (work rolls) for multi-roll stands, gauges, and smaller plastic molds, which require high wear resistance, are the most common applications of K110. [24].

The usual heat treatment condition of the steel can be seen in Table 3.

Table 3. Usual heat treatment conditions of the as-delivered material [24]

Annealing	Stress relieving	Hardening	Continuous cooling	Tempering
√	√	√	√	√

Physical and mechanical properties are important to know during designing tool steels since reliable and safe operations are determined by some of these properties. Some thermal electrical and mechanical properties of the K110 base material can be seen in Table 4.

Table 4. Selected physical and mechanical properties of K110 steels [24]

Properties (at 20° C)	Unit	Value
Density	kg/dm ³	7.67
Thermal Conductivity	W/mK	23.90
Specific Heat	J/kgK	470
Special electrical resistance	$\Omega \cdot \text{mm}^2/\text{m}$	0.65
Modulus of elasticity	GPa	200

3.1.1. Sample processing

Sample cutting from the steel rod. The initial diameter of the rod was 50 mm, but after the cutting process, we got 30 mm as a final diameter, as required by the tribological test samples. To avoid residual stress, it is important to cut the rod from the first attempt.

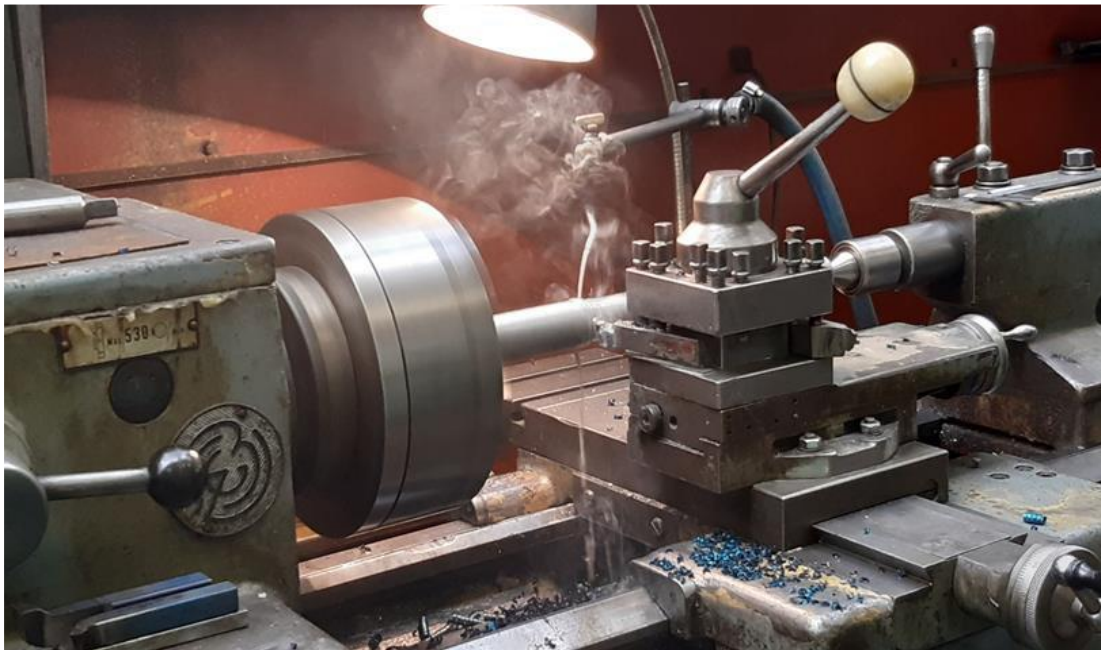


Fig. 10. Cutting process

Grinding process of the steel rod. As a next step, we used the grinding machine for making the surfaces of the rod parallel (Fig. 11.). Grinding employs an abrasive product, usually, a rotating wheel is brought into controlled contact with a work surface. The grinding wheel is composed of abrasive grains held together in a binder. These abrasive grains act as cutting tools, removing tiny chips of material from the work. Most abrasives used in industry are synthetic. Aluminum oxide is

used in three-quarters of all grinding operations and is primarily used to grind ferrous metals. The fixing table of the grinding machine is electromagnetic.



Fig. 11. Grinding process

Sawing process. With this equipment, we cut the steel rod into little pieces, creating the test pieces for the planned different test procedures (Fig. 12.).



Fig. 12. Sawing machine

Grinding and drilling process on specimens.

After the heat treatment process, we did the grinding process on the front surfaces of the specimens. We've already described the most important features of a grinding process.

The two grinding operations differ in that the first case involves cylindrical grinding, during which the diameter of the rod is specified in the diameter direction,

while in the second case, the front surface of specimens is plain ground. The aim of the latter one is to make the quality of the surfaces as fine as possible, on the other hand, to make the surfaces, which will be used as a basis and test surface during tribological testing, as parallel as possible.

The next step was making a hole in the specimens (Fig. 13/a). It will be helpful in heat treatment processes for hanging the specimens in the furnace. We did this operation in the workshop with the help of the master. The final appearance of the specimen is shown in Fig. 13/b.

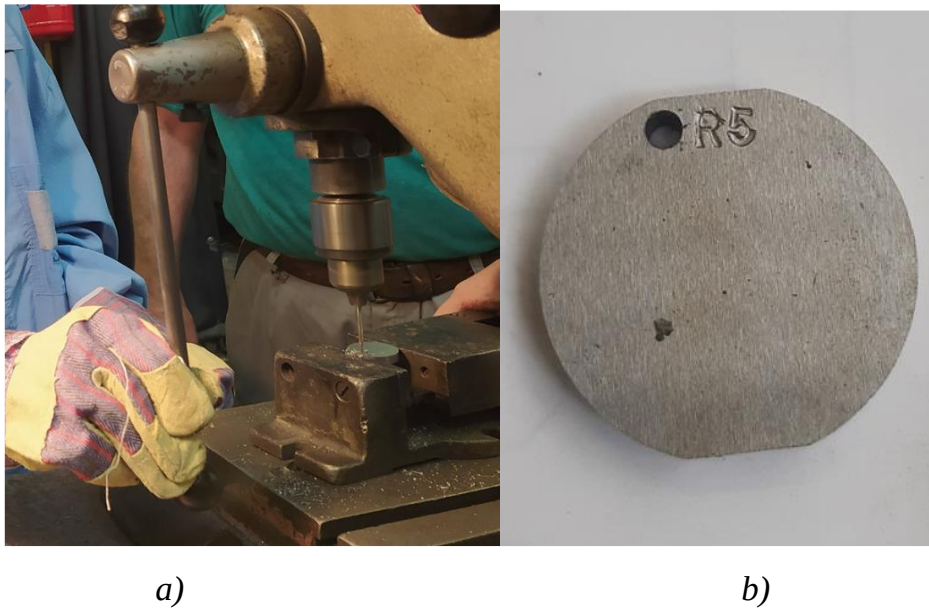


Fig. 13. Drilling process (a); and the final appearance of the specimen (b).

3.1.2. Heat treatment

A 3-stage heat treatment process, called precipitation hardening were applied on our specimens, to provide high strength of the tool material. This is necessary on one hand to assure acceptable mechanical loadability of the tool, on the other hand, to provide appropriate support for the hard ceramic coating. The stages of the heat treatment process are as follows:

1st stage. Heating

- 1) $T=530\text{ }^{\circ}\text{C}$, holding time=0.5 hour
- 2) $T=850\text{ }^{\circ}\text{C}$
- 3) $T=1060\text{ }^{\circ}\text{C}$, holding time=25 min

2nd stage. Quenching

Cooling in oil

T=150-200 °C to avoid cracks, holding time=3-4 min

3rd stage. Tempering

- 1) *T=540°C, holding time=3 hours;*
- 2) *T=540°C, holding time=2 hours;*
- 3) *T=540°C, holding time=2 hours.*

The reason for the three steps during heating is that the heat conduction of highly alloyed steel is very low. Thus, high thermal stresses may arise during quick heating. The steps contain also holding periods providing heat to be equalized in the material in order to avoid cracking.

Each tempering process is followed by air cooling in half a day. The samples were anointed by “FEKECS” before heat treatment to avoid oxidation, formation of clinker, etc. The “FEKECS” called blackwash (or greensand) can be a mixture of graphite, sand, water, and linseed oil, but it can also be silicate powder slurries suspended in alcohol (methylated spirit). For our specimens, these materials are used to form a protective layer during high-temperature treatments.



Fig. 14. Specimens in the furnace

3.1.3. Polishing

Polishing process:

The final step in creating a flat, scratch-free, and mirror-like surface is polishing. Abrasives suspended in a water solution are applied to a cloth-covered electrically driven wheel for polishing.

The properly polished specimen should have a mirror-like surface free of scratches after 1 or 2 minutes. Since the napped material will adhere to the specimen shape

under pressure, minimal pressure should be applied during final polishing, and time should be held to a minimum.

The wetness of the final polishing cloth has a significant impact on the final product. If the clothing is too muddy, the sample will show pits; if it is too dry, the sample will buff and/or smear. The sample should be removed from the wheel and the time taken for the polishing film to dry (five to eight seconds) should be tested to determine the proper wetness. A thin opaque film indicates that there is enough abrasive.

Highly fine grades of diamond abrasives are often used for the final polishing sequence in precision work. Cerium Oxide is especially excellent for the final polishing of aluminum and other soft metals and alloys [25].

3.1.4. Coating process

As a next step the coating process for the samples were done by our industrial partner Oerlikon Balzers Coating Austria GmbH Magyarországi Fióktelepe (Balzers GmbH Mft.), Székesfehérvár, Hungary. The technological process of coating involves generally the following steps.

- Incoming. Definition of the specific workflow and inspection of the incoming goods.
- Cleaning. Defining and accomplishing the cleaning method, followed by visual inspection.
- Pre-treatment: Optional pre-treatment of parts with blind holes or a critical surface
- Degassing
- Microblasting
- Wetblasting
- Loading. This stage begins with placing the parts into fixtures. Then, assembly the loaded spindle on the carousel. After that, they install the test parts and check the rotation of fixtures.
- Coating. This step involves also various subprocesses like pumping, heating, etching, deposition of the coating material, cooling, and flooding, under accurate control of the process.
- Unloading: After the coating stage is done, they remove parts from fixtures and send parts to final inspection.

- Post-treatment. Optional post-treatments can be applied automatic or manual brushing, microblasting or wetblasting, and drag finish
- Inspection: Final inspection is accomplished to determine the hardness (HRA, HRC), the coating adherence (HRC, HRA), the coating thickness (calotest grinding, XRF), and magnetism. Then a visual inspection is applied.
- Preservation: The preservation process is done by oil, emulsion, or VCI medium (dry)
- Forwarding: After all these stages, the parts are packaged and prepared to forward the delivery point.

Our specimens are made by the Advanced coatings of Formera and Lumena.

Formera is used when you want to ensure high productivity when forming high-demanding steels, especially when resilient coating solutions are required. We can specify it like:

- Up to 680% more efficient than comparable coatings
- Great abrasive wear protection
- Excellent coating adhesion

The recommended application for the Formera are:

- Tough draw and flanging applications >500 MPa 1.5mm
- High strength steel applications
- Deep drawing of AHSS
- Flanging of AHSS
- Trimming of AHSS [26].

The extremely high hardness of Lumena fulfills the high demands and is ideal for injection moulds for GF and CFK reinforced plastics, moulds made of steel with low hardness (< HRC 40), and for forming high strength metal sheets. These properties are characteristic for the Lumena:

- Extreme high hardness
- Excellent wear protection
- Improved plastic flowability
- Increases part quality

The recommended applications for the Lumena are:

- Plastic injection moulding (moulds with textured surfaces, processing of GF and CFK reinforced plastics)
- Moulds for plastics processing made of steels with low hardness (> 40 HRC)
- Forming of high strength metal sheets
- Die casting applications [27].

Both of the coatings are made at $480\text{--}500^\circ\text{C}$, and theoretically, they have 6-9 microns thickness. They are made by one of the PVD-based processes is called arc evaporation. In this process, an arc with a diameter ranging from microns to just a few tenths of a micron is run over the solid, metallic coating material, causing it to evaporate. The high currents and power densities used to cause almost complete ionization of the evaporated material and, thereby, the formation of high-energy plasma. The metal ions combine with a reactive gas that is introduced into the chamber, strike the target tools or components with high energy, and are deposited as a thin and highly adherent coating [28]. The characteristics of coatings are described in Table 5.

Table 5. Characteristics of the Formera and Lumena coatings (informations are taken from the producer's homepage, relevant for products in general) [29]

Characteristic	Formera	Lumena
Coating material	CrAlN	TiAlN
H _{IT} hardness of coating, GPa	28+/-2	33+/-3
Friction coefficient against steel (dry)	0.35	0.3-0.35
Usual thickness of coating, μm	6-12	8-12
Residual stress, GPa	compressive stress -2+/-0.5	compressive stress -2+/-1
Max. operation temperature, $^\circ\text{C}$	900	900
Coating temperature, $^\circ\text{C}$	480	450
Colour of coating	silver-light grey	violet-grey
Structure of coating	nano-structured	mono-layered
Available in advanced version	√	√

3.1.5. Microstructure of the coated samples

The photos (Table 6., Table 7.) illustrate the microstructure of the precipitation-hardened base material (the steel substrate) with several II. phases which are hard carbides or intermetallics precipitations in the metallic matrix. They are arranged visibly in series (queuing) the reason for which is as follows: the steel base material from which the discs were cut was produced by rolling. This cold forming operation (rolling) of the bar results in a texture of the metallic material, and then the precipitates were arranged in series in the base material during heat treatment of the discs.

Table 6. Cross-section microscopic photos of Formera coated sample, magnification: 100 $\times$, 500 $\times$

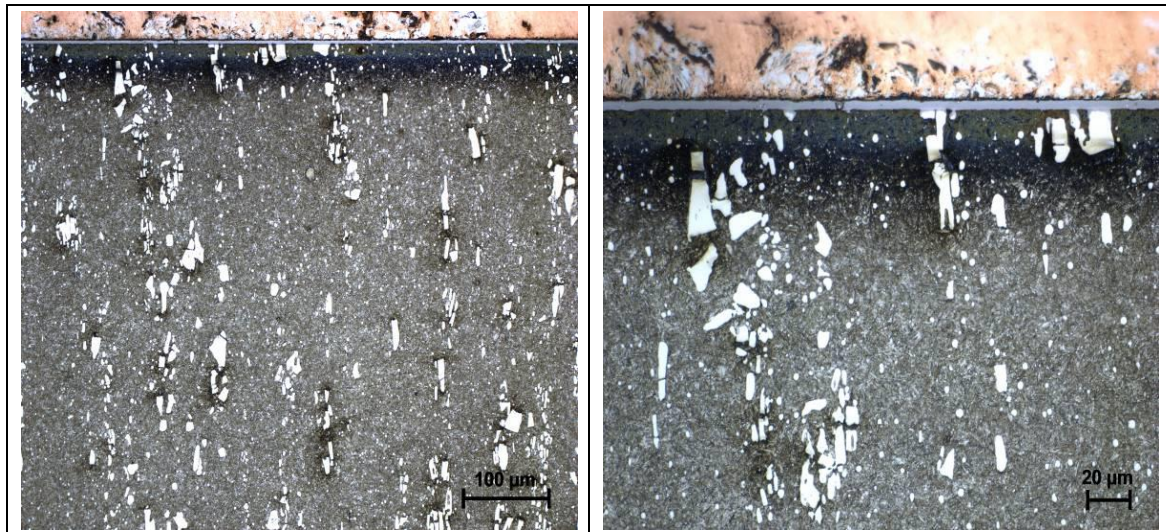
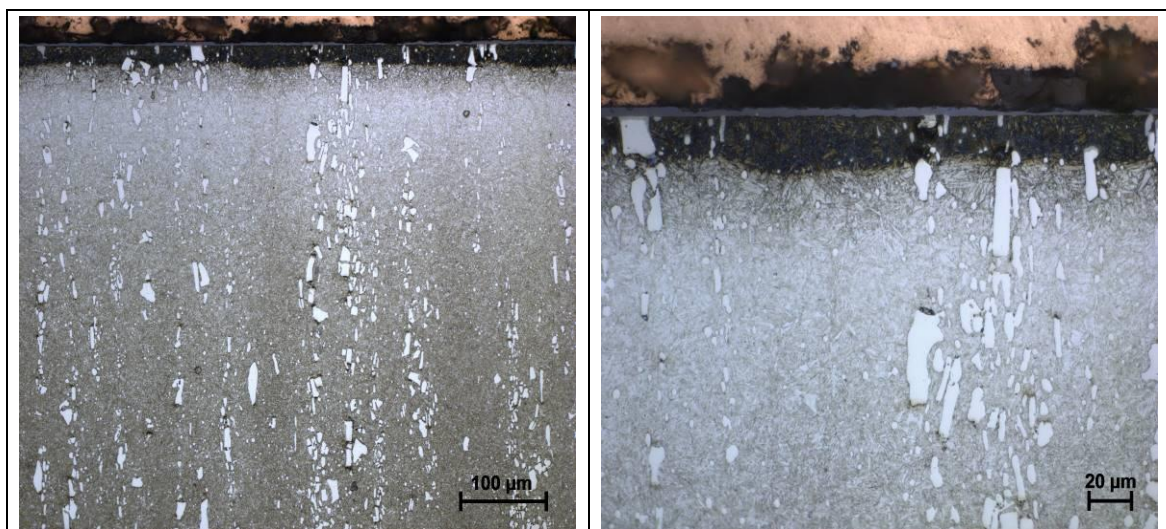


Table 7. Cross-section microscopic photos of Lumena coated sample, magnification: 100 $\times$, 500 $\times$



3.1.6. Surface preparations

A specific step-by-step method must be followed when preparing metallographic specimens to assess microstructure and material. To expose correct microstructures, specimens must be kept clean and preparation procedures must be carefully followed [25].

Grinding process:

The specimen must be free of scratches, stains, and other surface flaws, preserve nonmetallic inclusions, show no signs of chipping due to brittle intermetallic materials, and phases, and be free of any traces of disturbed metal [25].



Fig. 15. Grinding machine



Fig. 16. Polishing equipment

Etching process:

Only a few structural characteristics such as inclusions, holes, or other physical imperfections can be visible under a microscope in a properly polished, unetched specimen. Etching is used to draw attention to, and often identify, microstructural features or phases.

Because of the differences in the rate of attack of the different phases present and their orientation, etching occurs when acid or base is applied on the specimen surface.

Typically, the etching process is completed by simply adding the required solution to the specimen surface for a few seconds to several minutes.

The etchant of choice for traditional irons and steels is Nital, a Nitric Acid-Alcohol mixture. Using an eyedropper or cotton swab, Nital is dripped onto the specimen. Depending on the sample and nital concentration, ten seconds to one minute is normally enough for proper etching.

After washing the sample under running water, rinsing it with alcohol, and drying it in an air blast, the microscopic analysis stage begins. [25].



Fig. 17. Tools and accessories for the etching operation

4. TEST RESULTS AND EVALUATION

4.1. Hardness test

The application of hardness testing enables to evaluate the properties of a material, such as strength, ductility and wear resistance, and so helps to determine whether a material or material treatment is suitable for the required purpose. We did 5 measurements with the Vickers hardness tests for each type of coatings and for the uncoated material, applying 0.5 N and 1 N loads for our samples. The obtained average hardness values are presented in Table 8., while Figure 20. visualize these values in a diagram providing the easy comparison.

Table 8. Hardness test results

Coating	Loading force, N	HV 0,05; HV0,1							
		1	2	3	4	5	Average	SD	Var. coeff., %
Uncoated (Reference)	0.5	536	521	515	532	528	526	8.4	1.6
	1	547	528	534	520	530	532	9.9	1.9
Lumena	0.5	1607	1855	2381	2185	1787	1963	313.6	16.0
	1	1736	1493	1601	1435	1380	1529	141.7	9.3
Formera	0.5	2125	1714	1900	2403	2079	2044.2	258.0	12.6
	1	1922	1850	1911	1810	1823	1863.2	50.9	2.7

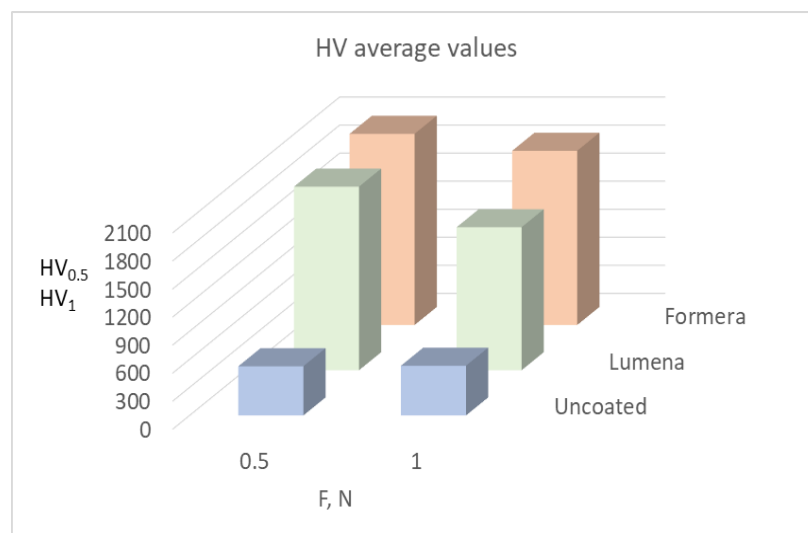


Fig. 18. Diagrams representing the results of the hardness test, done with 0.5 N and 1 N loads on the uncoated samples, Lumena coated and Formera coated samples

As we can see, in the case of both applied loading forces, the Formera showed higher hardness values and both coating hardnesses are at least 3 times greater than that of the reference material.

4.2. Calotest (ball cratering test)

The test was accomplished by a 30 mm diameter steel ball, using alumina abrasive suspension.

After making the worn indentation by this Calotest procedure, we determined the outer and inner diameter of the indentation by optical microscopic investigation as described in section 2.4.3.

Then we calculated the thickness of the layers using the equation (1):

$$s = T - t = \frac{1}{2}\sqrt{4R^2 - d^2} - \frac{1}{2}\sqrt{4R^2 - D^2} \quad (1)$$

where:

- s is the thickness of the coating layer,
- T is the total depth of the worn track,
- t is the total depth of the worn track in the substrate,
- R is the diameter of the ball,
- d is the smaller diameter, representing the worn diameter in the substrate (measured), and
- D is the bigger diameter, i.e. the total diameter of the worn track (measured).

The worn indentations are shown in Fig 21. and Fig 22.

It can be seen, that the first indentation was significantly bigger on both specimens and the following ones were smaller and smaller. Here it should be noted that this phenomenon does not affect the calculation results, because the ratio of the diameters is considered by the equation.

The numerical results obtained by the Calotest are summarized in Table 9. and Table 10. Based on this test, we defined the average layer thickness of Fomera coating as 5.77 µm, and Lumena coating as 4.7 µm, concluding that Formera coating thickness is about 20% higher than that of the Lumena.

The difference should be considered during the evaluation of the other tribological test results.

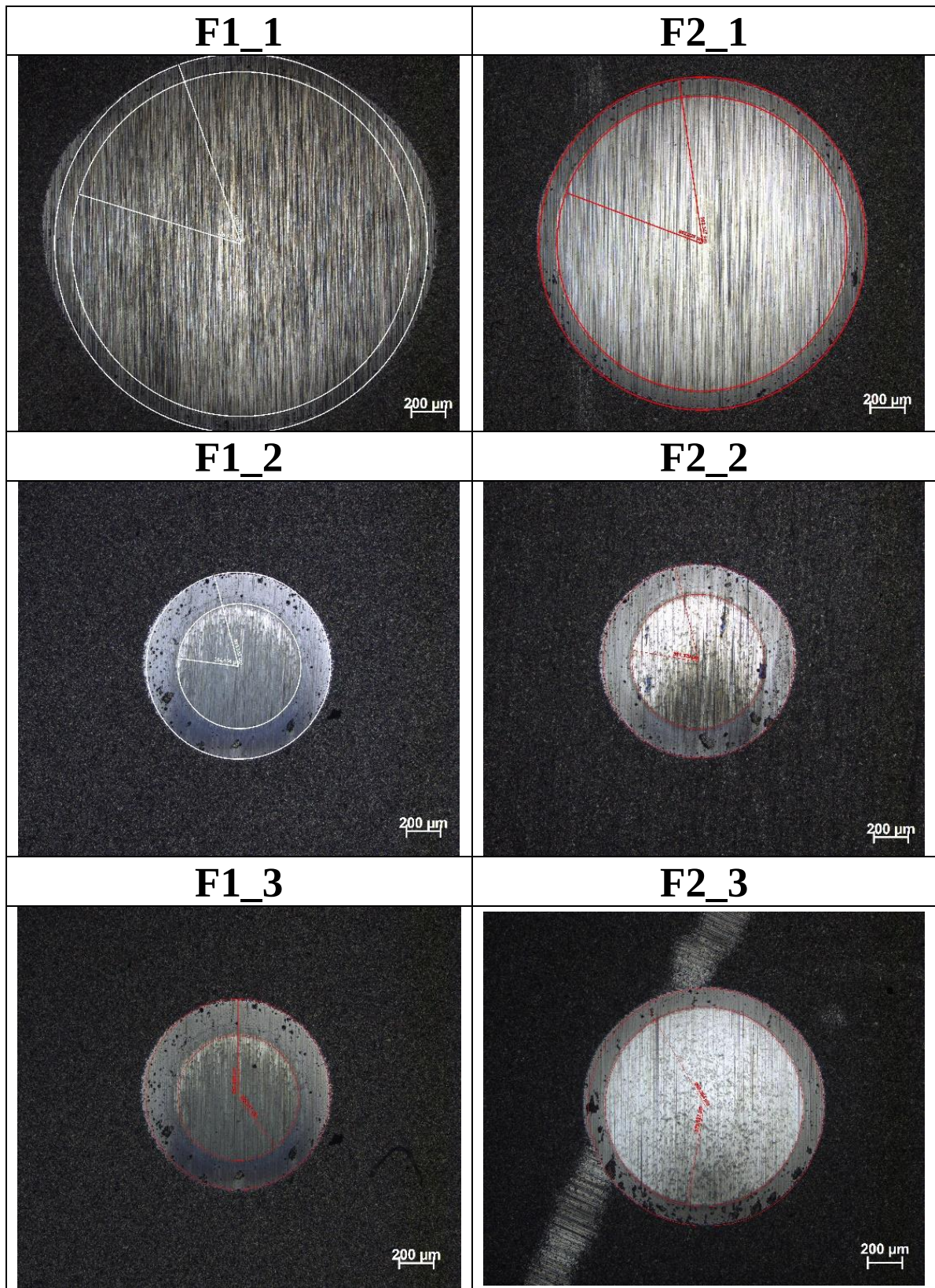


Fig. 19. The microscopic images of the worn tracks in the Formera coating after the Calotest

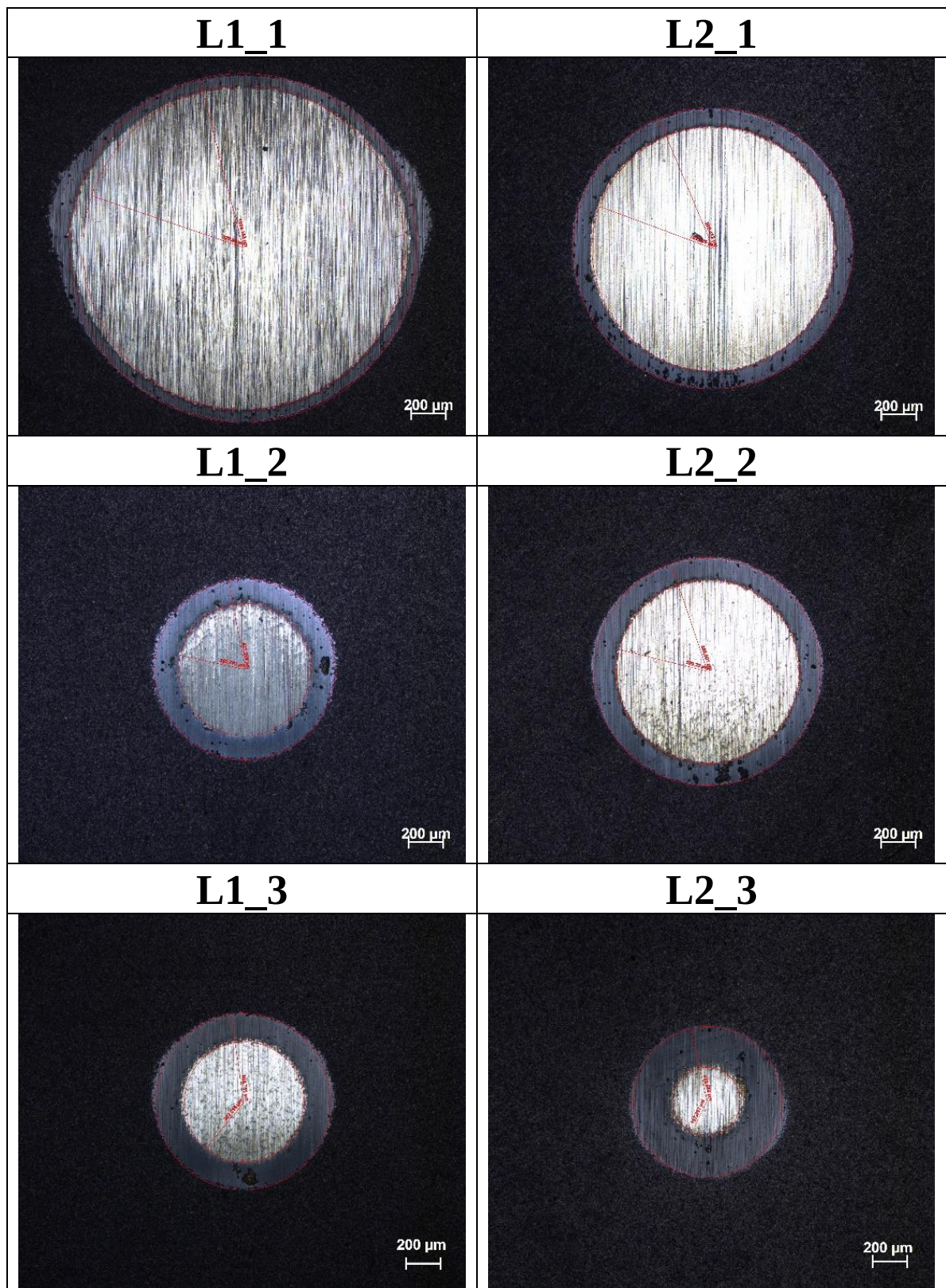


Fig. 20. The microscopic images of the worn tracks in the Lumena coating after the Calotest

Table 9. The measured thickness values on the Formera coating

Sample mark	Thickness of the FORMERA coating, μm	Average, μm	St. dev. μm	Var. coeff., %
F1_1	6.97	5.77	0.87	15.11
F1_2	5.41			
F2_1	6.65			
F2_2	5.43			
F1_3	5.47			
F2_3	4.66			

Table 10. The measured thickness values on the Lumena coating

Sample mark	Thickness of the LUMENA coating, μm	Average, μm	St. dev. μm	Var. coeff., %
L1_1	4.77	4.80	0.39	8.11
L1_2	4.18			
L2_1	5.30			
L2_2	5.02			
L1_3	4.55			
L2_3	4.96			

Considering the information obtained from the producer, the measured coatings thickness values, shown in Table 9. and Table 10. were systematically lower than the related characteristic coating thickness values presented in Table 5. In the case of the Formera coating, the difference between the measured average ($5.77 \mu\text{m}$) and the lower limit given by the producer ($6 \mu\text{m}$) is negligible, however, in the case of the Lumena coating the measured average value ($4.80 \mu\text{m}$) is significantly lower than the characteristic lower limit of this kind of coating ($8 \mu\text{m}$), given by the producer.

4.3. Scratch test

Scratch testing is carried out to get an insight into materials to determine the resistance to abrasion and wear of modern composite materials and automotive paints varnishes, as well as wear resistant coatings. we have already described the principle and accomplishment of the process in section 2.4.4

The SP-15 uniquely developed scratch tester equipment is shown in Fig. 21.

We performed instrumented scratch tests for both coating types by using constant and increasing loading force, as well. A detailed description of the executed experimental procedure, the characteristic test recordings, and result evaluation is given separately for the two types of loading in the next sections.



Fig. 21. The SP-15 instrumented scratch tester at the laboratory of IMST, UM

4.3.1. Increasing loading scratch test

First, we performed the increasing load scratch tests to find the critical loading force causing the delamination of the coating and to analyse the damage process of the coatings under progressive loading.

The parameters of the increasing load tests are shown in Table 11.

Table 11. Testing parameters for increasing load scratch tests

Parameter	Nomination	Value
Starting value of normal force	$F_{n,min}$	2 N
Ending value of normal force	$F_{n,max}$	130 N
The gradient of the normal force	dF/ds	10 N/ mm
Length of scratches	L	13 mm
Velocity of indenter	v	5 mm/ min
Indenter	-	HRC indenter

The photos of coated samples (Formera and Lumena) with increasing load scratches are described in Fig. 22.

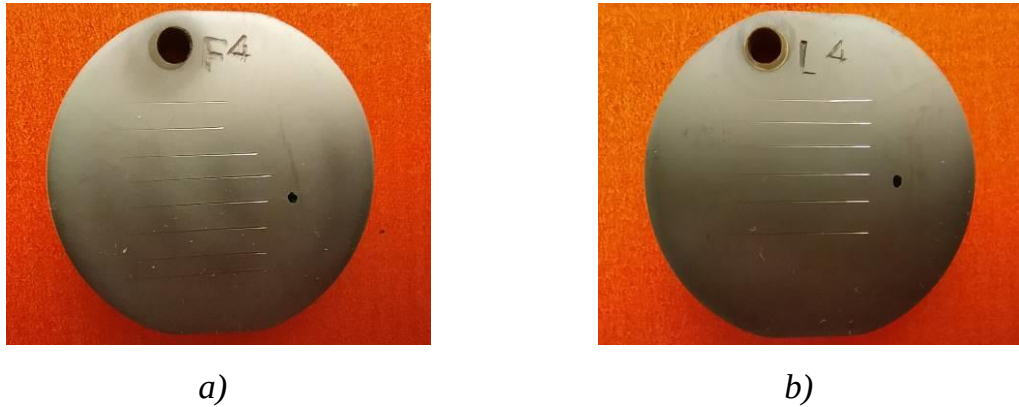


Fig. 22. The photos of coated sample Formera (a) and Lumena (b) with scratches produced by increasing load

To observe the damage mechanism of the different coatings, we took low magnification panorama photos of the scratches (Appendix 1), two of which are shown in Fig. 23.

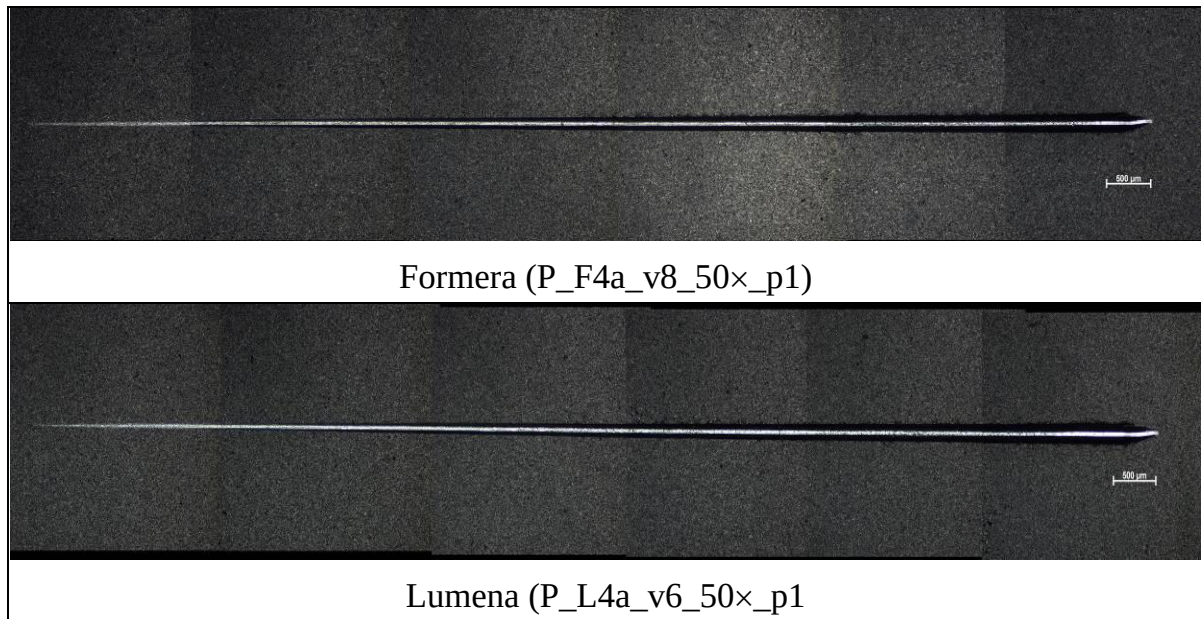
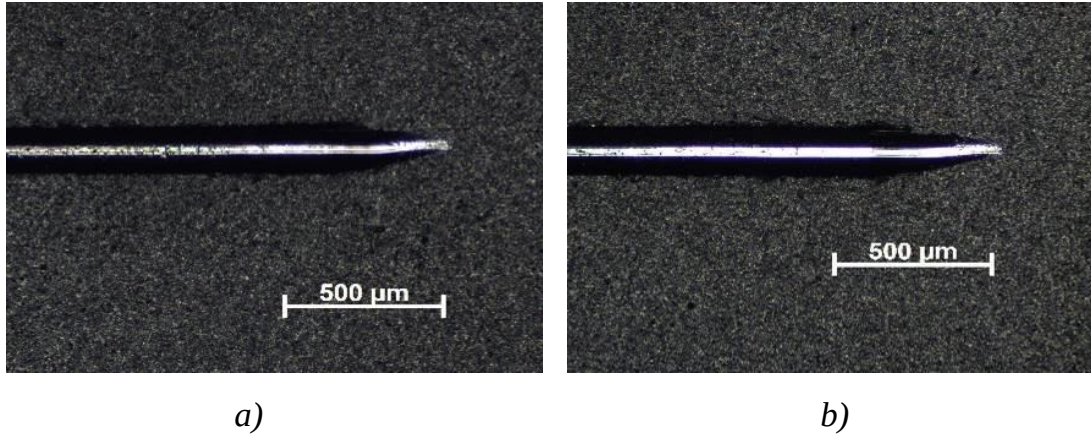


Fig. 23. Panorama photos of scratches produced by increasing loading force tests on Formera and Lumena coated samples

Detailed observation of the magnified last item of the panorama photos (Fig. 24.) reveals important differences in the last section of the scratches. In spite of the very low magnification and weak visibility of the picture, it is clearly seen that the whole delamination of the Formera coating (Fig. 24 – a.) starts at a much longer distance compared to that of the Lumena coating (Fig. 24 – b.). While for Formera coating

this distance is $L_{crit} = 12.7$ mm, for Lumena coating it is $L_{crit} = 11.8$ mm (see scratch diagrams denoted by F4a_v8 and L4a_v6 in Appendix 1.1 and Appendix 1.2).



$$F_{crit, Formera} = 124 \text{ N (Scratch: F4a_v8)} \quad F_{crit, Lumena} = 112 \text{ N (Scratch: L4a_v6)}$$

Fig. 24. Comparison of the scratch morphology in the vicinity of the critical loading force for Formera (a) and Lumena (a)

The individually measured critical loading forces are presented in Table 12.

Table 12. The measured F_{crit} forces for Formera and Lumena coatings

Coating	Scratch identifier	F_{crit} , N	Average	SD, μm	Var. coeff., %
Formera	F4a_v3	106	114.8	5.71	4.97
	F4a_v4	115			
	F4a_v5	114			
	F4a_v6	115			
	F4a_v7	115			
	F4a_v8	124			
Lumena	L4a_v1	90	105.7	8.07	7.63
	L4a_v2	108			
	L4a_v3	111			
	L4a_v4	105			
	L4a_v5	108			
	L4a_v6	112			

Observations from the progressive loading scratch tests:

- The average critical normal load for Formera coating is 115 N, while this value is 106 N for the Lumena coating. These critical normal loads represent the loading at which the total delamination of the ceramic coating starts. This value is almost 9% higher for the Formera coating. The variation coefficient – in the case of both types of coating – is less than 8%, thus the reliability of the measured critical loading force values are quite good.

- From the point of view of the reliable and safe operation of the coated tools, different damage processes, i.e. minor injuries of the coatings that are caused by lower loads – i.e. by the so called subcritical loads –, can also be useful to define. Unfortunately, in the framework of this research work, due to the COVID restrictions, preventing me to use the laboratory equipment in the last period of the work, we had no time for such detailed analysis of the panorama photos. Such analysis requires taking several further microscopic images about the different regions belonging to the subcritical loadings.

If we examine the characteristics of the individual curves of the coatings in the scratch diagrams, we can recognize further important features of the different scratch behaviour of the coatings. Fig. 25. illustrates one-one characteristic curve for Formera and Lumena in one diagram, based on which we drew the following conclusions:

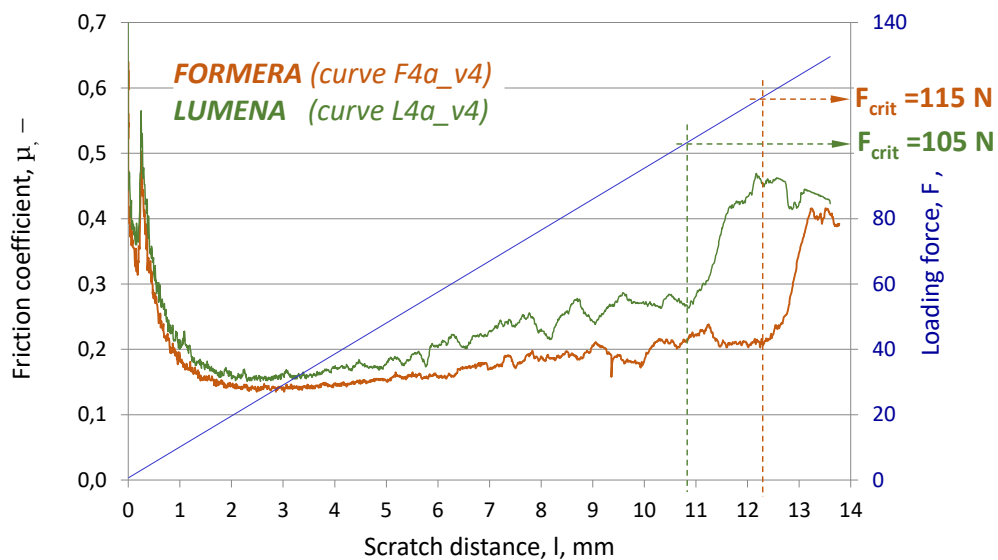


Fig. 25. Comparison of the scratch behaviour of the Formera and Lumena coatings based on two characteristic curves taken during progressive loading scratch tests

- With increasing the normal load the friction coefficient curves rise less steeply for the Formera coating, as compared to those for the Lumena coating.
- Taking into consideration that Formera coating possesses higher hardness, than the Lumena coating, we suppose that the Lumena coating is probably more brittle, has higher crack sensitivity, i.e. it more readily damages during scratching. Thus the higher amount of debris in the path of the scratch instrument increases the coefficient of friction.

- Further morphological analysis of the wear debris, by higher resolution microscopic technique (e.g. by SEM), would provide the possibility for deeper analysis of the differences in the damage mechanisms of the coatings. Such kind of analyses are planned to be accomplished in the framework of our future research work in this area.

4.3.2. Constant load scratch test

To investigate the coating behaviour at subcritical loads we accomplished constant load scratch tests at two different loads, below the critical loading force regarding both investigated coatings. The whole set of the progressive loading scratch test diagrams is found in Appendix 2. Based on the results of the increasing load scratch diagrams, we have determined the applied constant loads with the following considerations: at $F = 50$ N loading force the Formera coating is expected to show no significant damage, while the Lumena coating will probably show some initial failure of the coating. In contrast, at $F = 80$ N, both coatings are expected to show visible failure. Accordingly, we chose the two constant loads to be $F = 50$ and 80 N.

The test parameters for the constant load scratch tests are described in Table 13.

Table 13. Testing parameters for constant load

Parameter	Nomination	Value
Constant load	F_n	50 and 80 N
Length of scratches	L	6.6 mm
Velocity of indenter	v	5 mm/ min
Indenter	-	HRC indenter

The photos of coated samples (Formera and Lumena), containing scratches taken by constant load, are shown in Fig. 26.



Fig. 26. The photos of coated sample Formera (a) and Lumena (b) with constant load scratches.

The whole set of the diagrams obtained during the constant load scratch tests are contained in Appendix 3.1-3.3., and the individual numerical results for the friction coefficient values are found in Appendix 3.4.

Table 14. Friction coefficient values obtained from constant load scratch tests

Coating type	File name	Constant load, F, N	Friction coefficient, μ			
			Measured	Average	SD	Var. coeff., %
Formera	F4b_v1_200x_2	50	0.157	0.156	0.001	0.641
	F4b_v2_200x_2		0.156			
	F4b_v3_200x_2		0.155			
	F4b_v4_200x_2	80	0.173	0.170	0.002	1.356
	F4b_v5_200x_2		0.169			
	F4b_v6_200x_2		0.169			
Lumena	L4b_v1_200x_2	50	0.175	0.168	0.004	2.571
	L4b_v2_200x_2		0.165			
	L4b_v3_200x_2		0.170			
	L4b_v4_200x_2		0.166			
	L4b_v5_200x_2		0.165			
	L4b_v6_200x_2	80	0.175	0.173	0.002	0.881
	L4b_v7_200x_2		0.172			
	L4b_v8_200x_2		0.173			

In Fig. 27. and Fig. 28. two representative diagrams related to the constant load scratch test for both coated samples are shown. The friction coefficient values were quite similar but systematically lower for Formera coating for both loadings. In case of $F = 50$ N constant loading the average was $\mu_{av,50,F} = 0.156$ for Formera, and $\mu_{av,50,L} = 0.168$ for Lumena coating, while in case of $F = 80$ N constant loading, these values were $\mu_{av,80,F} = 0.170$ and $\mu_{av,80,L} = 0.173$, for Formera and Lumena, respectively.

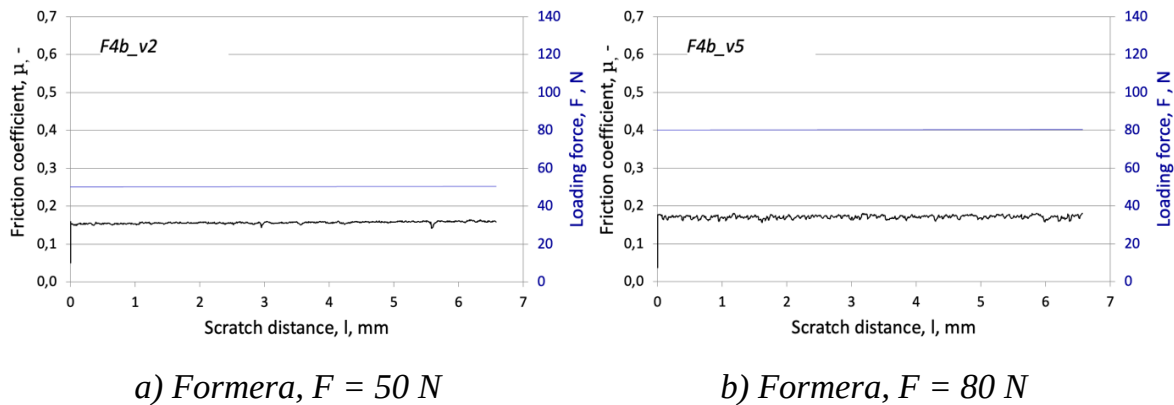


Fig. 27. Diagrams related to constant load scratch test for Formera samples.

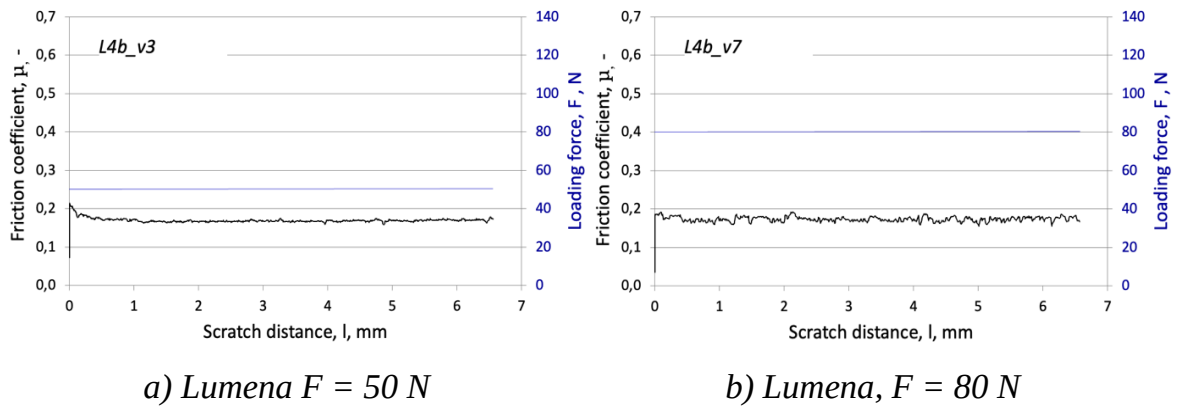


Fig. 28. Diagrams related to constant load scratch test for Lumena samples.

Optical microscopic pictures of the constant load scratches were analysed to measure the width of them. As the depth of field deteriorates during magnification of the optical microscopic investigations, we took two types of shots of scratch marks. In the first case, the microscopic pictures were taken by sharpening the scratch groove, in the second case, by sharpening the scratch contour to accurately measure the scratch width, as shown in Fig. 29.

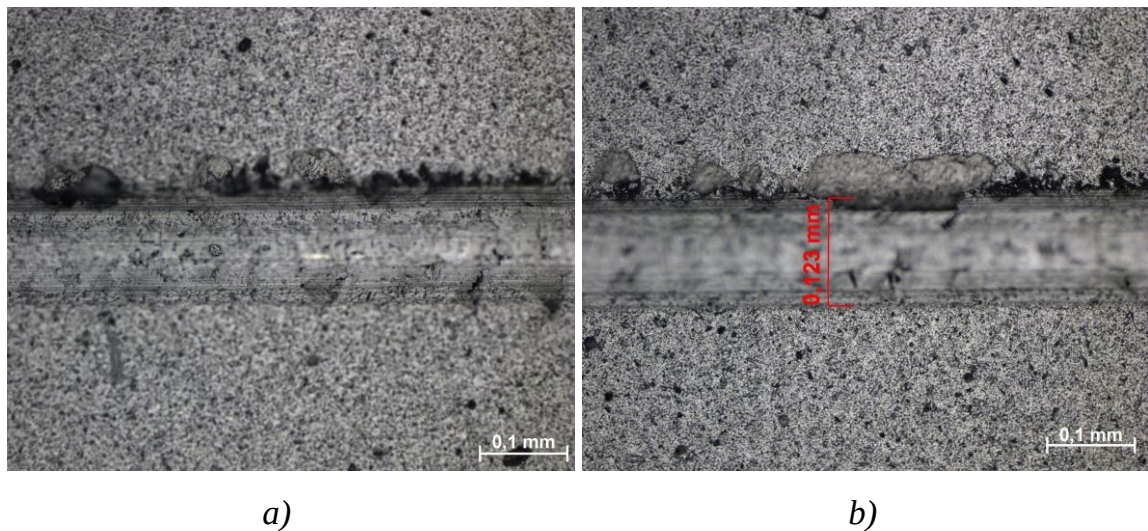


Fig. 29. Optical microscopic pictures of the constant load scratches; sharpening the scratch groove (a) and sharpening the scratch contour (b)
(Scratches are made on Formera coating: F4b_v5, $F=80 \text{ N}$)

For the qualitative analysis, the pictures sharpened for the scratch groove are shown in Table 15. and Table 16. Based on these optical microscopic images, we established that the scratch morphologies for $F = 50 \text{ N}$ loading were mostly similar, with some indication of the higher sensitivity of the Lumena coating to scratching, which is shown by the partial detachment of the coating. In the case of $F = 80 \text{ N}$ constant load both types of coating showed visible damages along the scratch

contour, but Lumena coating seemed to be more sensitive to scratching this case, as well, with the larger extension of chipping failures and delamination.

Table 15. Optical microscopic pictures of the constant load scratches; $F= 50\text{ N}$

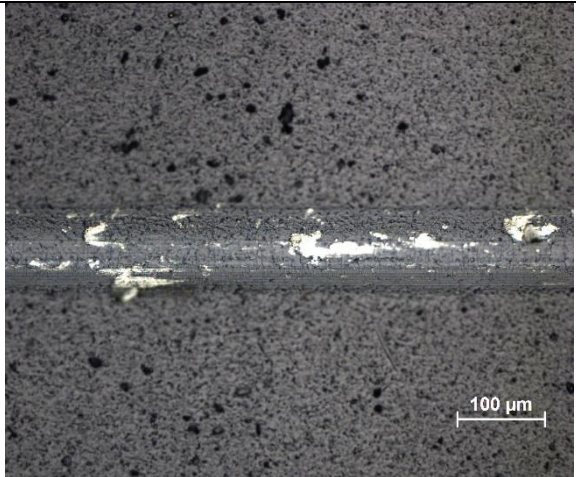
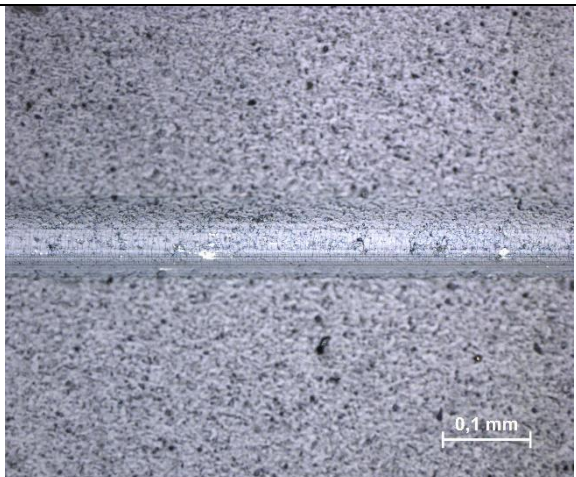
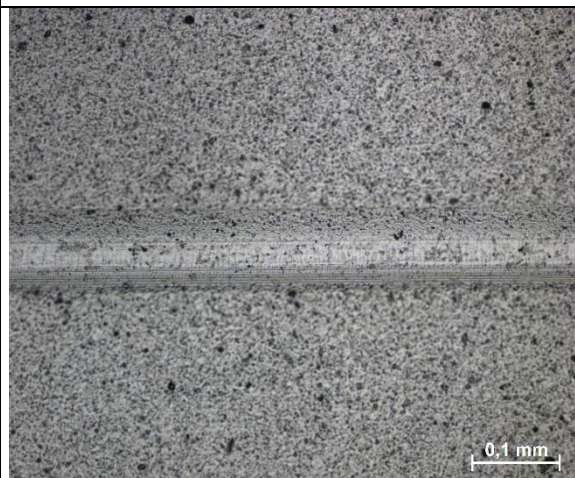
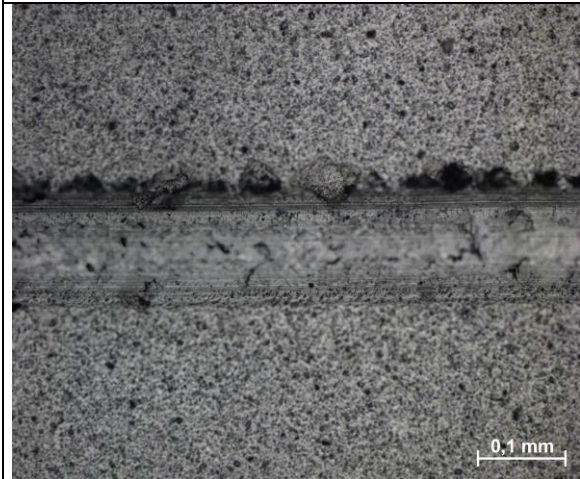
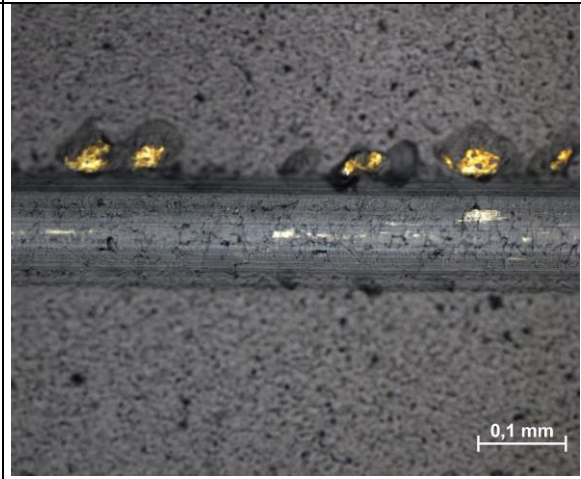
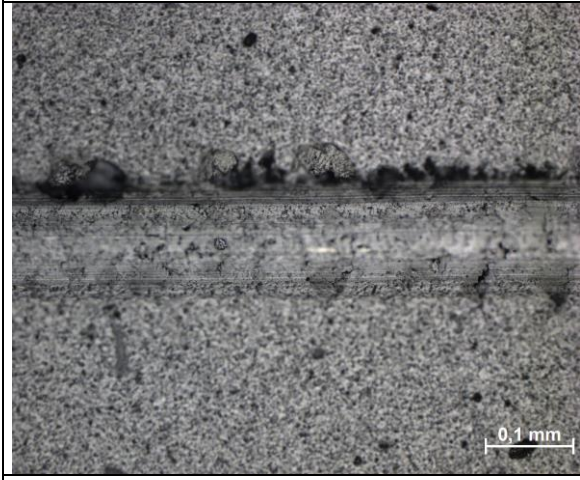
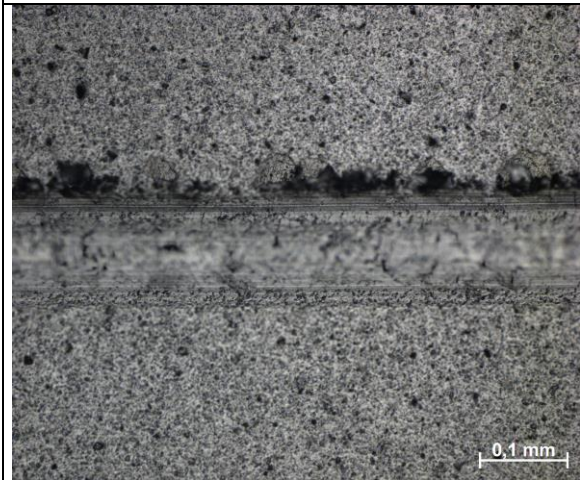
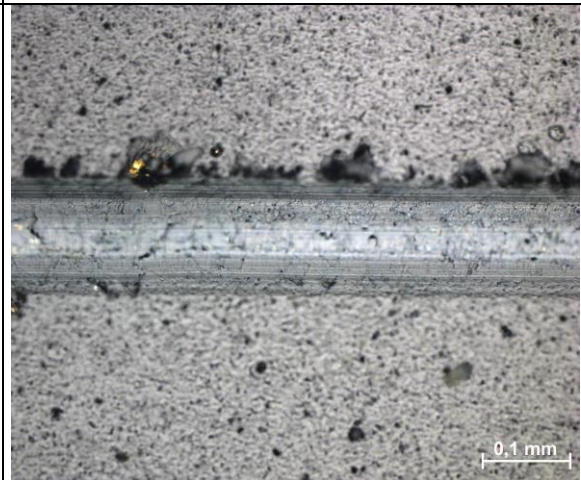
FORMERA	LUMENA
	
F4b_v1	F4b_v1
	
F4b_v2	F4b_v2
	
F4b_v3	F4b_v3

Table 16. Optical microscopic pictures of the constant load scratches; $F= 80\text{ N}$

Constant load: $F= 80\text{ N}$	
FORMERA	LUMENA
	
F4b_v4	L4b_v6
	
F4b_v5	L4b_v7
	
F4b_v6	L4b_v8

Here should be noted, that the aforementioned limiting conditions regarding the students entering the university laboratories, prevented me to finish this microscopic investigations, therefore the illustrations are the recordings of the preliminary tests, not made at the same scratch distance, so the damaging effect of the debris on the coating is not the same in each image. The optical microscopic pictures, illustrating the determination of the scratch width values, are presented in Appendix 4.1-4.3, while the numerical scratch width values, derived from these pictures are shown in Appendix 4.4. and in Table 17.

Table 17. Scratch width obtained from constant load scratch tests

Coating type	File name	Constant load, F, N	Scratch width, μm			
			Measured	Average	SD	Var. coeff., %
Formera	F4b_v1_200x_2	50	94	93.3	2.1	2.2
	F4b_v2_200x_2		91			
	F4b_v3_200x_2		95			
	F4b_v4_200x_2	80	128	124.3	3.2	2.6
	F4b_v5_200x_2		123			
	F4b_v6_200x_2		122			
Lumena	L4b_v1_200x_2	50	88	83.8	8.9	10.7
	L4b_v2_200x_2		71			
	L4b_v3_200x_2		91			
	L4b_v4_200x_2		78			
	L4b_v5_200x_2		91			
	L4b_v6_200x_2	80	118	120.3	2.5	2.1
	L4b_v7_200x_2		120			
	L4b_v8_200x_2		123			

Regarding the scratch width determined in this way, we can derive interesting conclusions from Table 17. The average of the measured scratch width values for the Formera coatings is somewhat higher, compared to that of the Lumena coating.

This finding is in contradiction with the formerly demonstrated results. We derived from the progressive load scratch test that the critical loading force, F_{crit} is higher for the Formera coating. In addition, we concluded from constant load scratch tests that the friction coefficient and damage sensitivity of the Formera coating is lower.

The question arises: how the scratch width for the Formera coating can be larger, indeed? Regarding that Formera coating possesses higher hardness and coating thickness, our final conclusions on the numerical scratch width results, are as follows:

- Scratch widths, determined by optical microscopy, were similar for both coatings but showed higher values in the case of the Formera coating.

- These scratch width values can not be considered as reliable quantitative results overall, due to the aforementioned uncertainties of the optical microscopic examination (depth of field problems).
- Therefore, a more accurate test method, such as profilometric studies is proposed to determine information on the scratch width and scratch resistance of the coatings.

Summarizing our findings during the progressive loading and constant loading scratch tests we conclude that the Formera coating possesses better resistance to scratch type loading, as compared to the Lumena coating.

Besides the scratch resistance, the wear resistance of the coating is also decisive from the point of view of the tribological behaviour. It can be determined by standard wear tests. In the next section, ball-on-disc tests, accomplished to characterize the wear resistance of the studied coatings, are described.

4.4. Ball-on-disc test

The ball-on-disc sliding wear test is a common tribological characterization method for determining the measure of the wear damage, the coefficient of friction and the dominant wear mechanism. The working principle of the test is already described in section 2.4.2. The equipment for executing a standard ball-on-disc test is shown in Fig. 30.

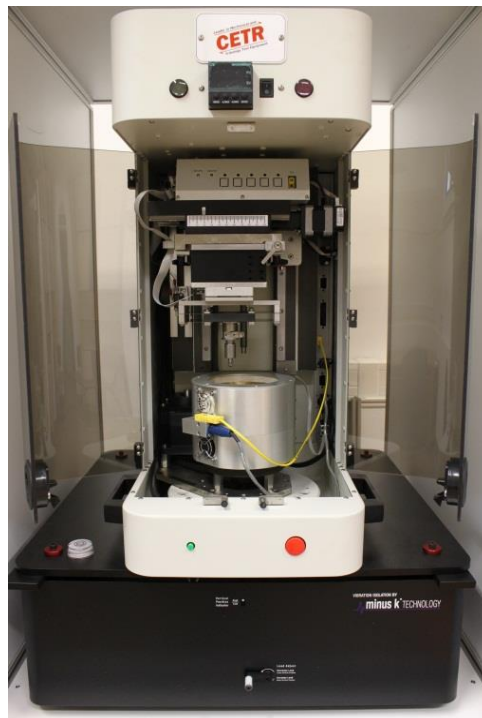


Fig. 30. The UNMT-1 surface tester with the tribological module

During the research work the ball-on-disc tests were accomplished using the following test parameters:

- applied loading force: $F=20\text{ N}$
- test time: $t=180\text{ min}$
- rotational speed: $n=95.542\text{ 1/min}$
- sliding speed: $v=30\text{ mm/s}$

The wear tests were executed in dry condition at an ambient air. The photos of the coated samples (Formera and Lumena) and reference material with wear tracks, are shown in Fig. 31.

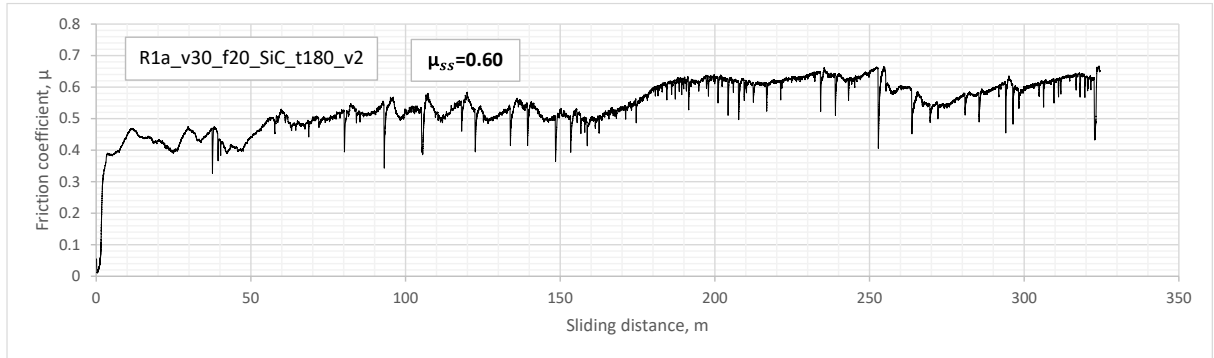


Fig. 31. Coated samples (Formera: top left, Lumena: top right) and the reference material (bottom) with wear tracks

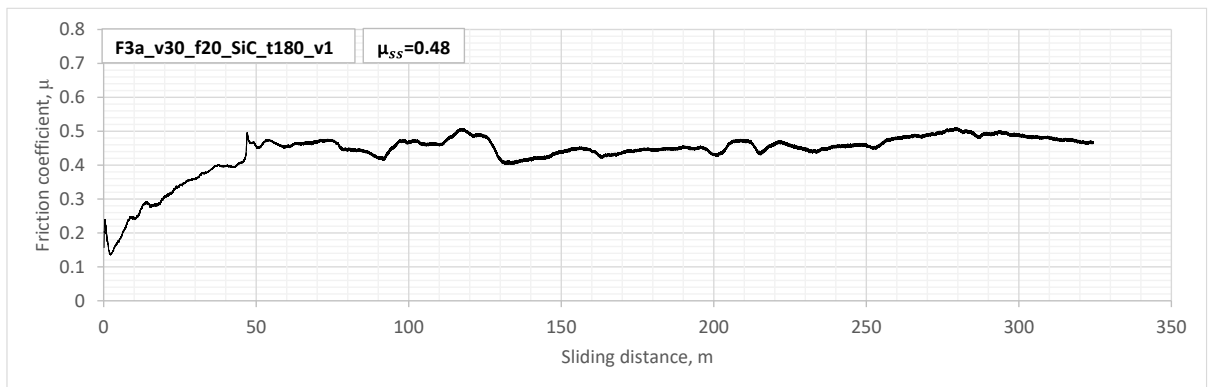
During the wear test accomplished on the sample of Lumena (L2), at the beginning, we heard a voice like the signal of loud squeaking but then it disappeared. Additionally, we heard a weird sound when we started the test on the sample L3, then it turned into a loud signal.

This experience is in harmony with the related friction coefficient diagrams, where the static friction coefficient, at the very beginning of the diagrams were very high for these two cases (0,57-0,75), indicating the strong sticking between the coated disc and the ceramic ball counterpart (see L2_1 and L3_1 diagrams in Appendix 5.).

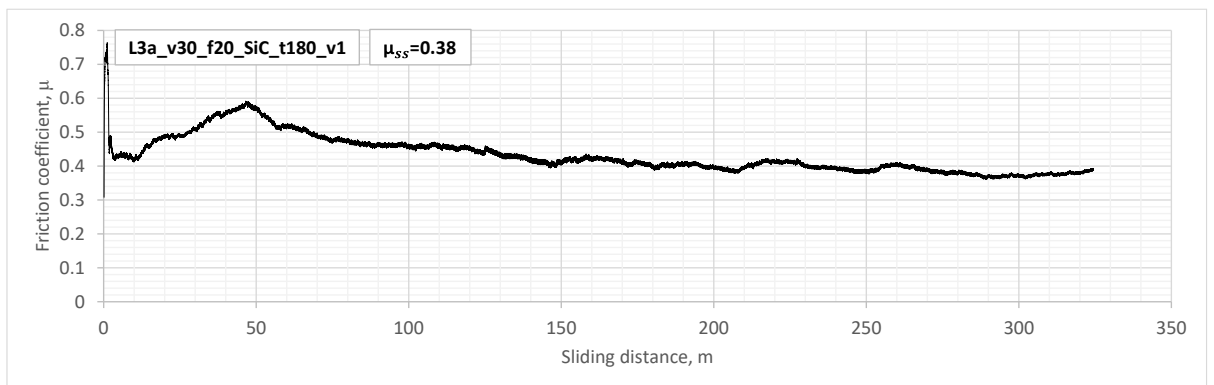
Typical friction coefficient diagrams of the coated and the uncoated reference material are shown in Fig. 32. The full collection of the related diagrams are contained in Appendix 5.1-5.3.



a)



b)



c)

Fig. 32. Characteristic friction coefficient diagrams recorded during ball-on disc test: a) Uncoated reference material; b) Formera coating; c) Lumena coating

Based on the ball-on-disc wear tests, we determined the steady state friction coefficient values as the average of those measured on the last 20% of the total sliding distance. These values for each tested case, with their average values and statistical features, are given in Appendix 5.4. and Table 18.

Table 18. The steady state friction coefficient values obtained from the ball-on disc tests

Coating type	File name	Steady state friction coefficient, μ_{ss} , –			
		Measured	Ave-rage	Stand. dev.	Var. coeff.,%
Formera	F3a_v30_f20_SiC_t180_v1	0.481	0.55	0.07	12.36
	F3a_v30_f20_SiC_t180_v2	0.617			
	F1a_v30_f20_SiC_t180_v5	0.556			
Lumena	L3a_v30_f20_SiC_t180_v1	0.381	0.44	0.07	15.31
	L2a_v30_f20_SiC_t180_v1	0.514			
	L2a_v30_f20_SiC_t180_v2	0.425			
Uncoated reference	R1a_v30_f20_SiC_t180_v1	0.53	0.57	0.04	6.33
	R1a_v30_f20_SiC_t180_v2	0.60			
	R2a_v30_f20_SiC_t180_v1	0.58			

The highest average coefficient of friction ($\mu=0.57$) was obtained on the uncoated reference material, while this value was $\mu=0.55$ for the Formera coating, and $\mu=0.44$ for the Lumena coating. To get a better description of the wear behaviour, we have measured the widths of the wear tracks created on the different samples. One-one characteristic wear track and the related measured width of the tracks are shown in Fig. 33.

For proper accuracy, we made the width measurements at four different locations on the wear track. The related microscopic pictures illustrating the wear track and the four sections with the measured values are found in Appendix 6.1.-6.3. The numerical results are summarized in Table 19. and in Appendix 6.4.

According to the width values of the measured wear tracks, compared to the uncoated reference material both coating types showed better wear resistance. The

smallest wear track width values were measured on the Formera coating (average 663.5 μm).

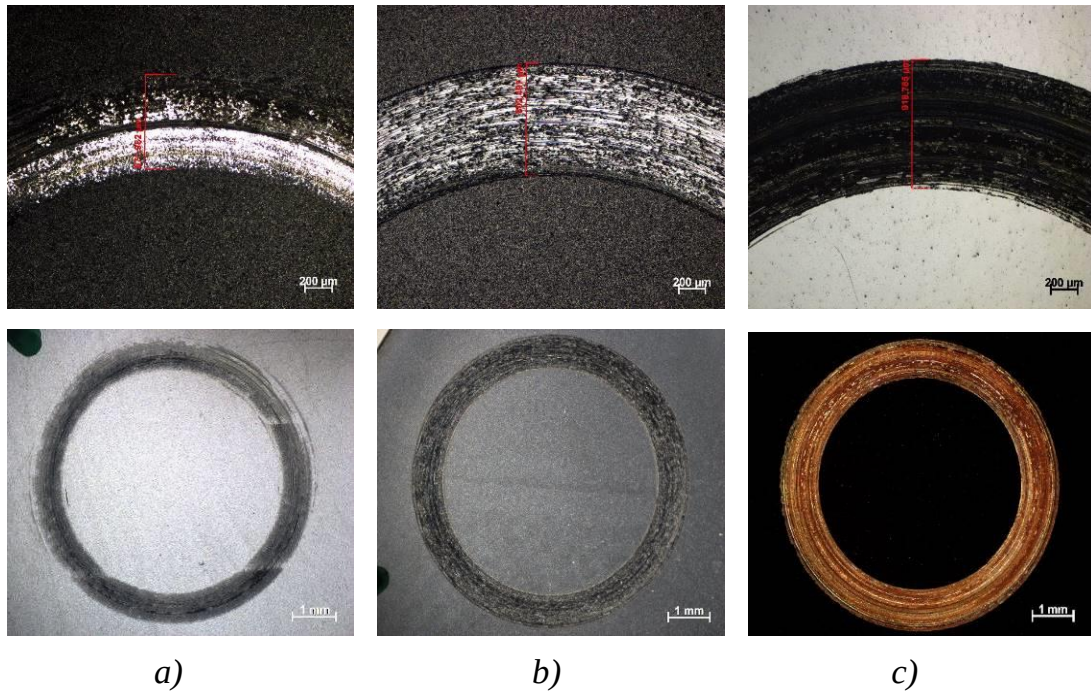


Fig. 33. Microscopic photos of wear width for a) Formera, b) Lumena and c) uncoated reference material

These results are generally in harmony with the scratch test results, excepting those obtained during the less reliable scratch width measurements when the size of the very thin, scratch grooves was not possible to measure with acceptable accuracy. Since the wear track was significantly wider than a scratch groove, the applied microscopic investigation provided more reliable results. In addition, we can improve the reliability of the wear damage evaluation by defining the worn cross section area with profilometry measurements, as well.

There is one more test result that should be discussed. The measured wear tracks' width values are not consistent with the friction coefficient values obtained from the ball on disc test for the two different coating types.

The friction coefficient values were lower for the Lumena coating and higher for Formera coating. At the same time, the Formera coating showed better wear resistance in terms of width measurements, i.e. while Formera showed a smaller damage tendency based on the wear track width, this behaviour was accompanied by a higher steady state friction coefficient, recorded during the wear test. The reason for this behaviour may be similar to that described in Section 4.3.2, i.e. the harder and thicker Formera coating seems to be more brittle and more sensitive for damage during friction, nevertheless, its wear resistance is still higher.

Table 19. Numerical values of wear track width obtained from ball-on-disc tests

Tested surface	File name	v, mm/s	F, N	Wear width, μm						
				Mea- sured	Average by track	SD, μm	var. coeff., %	Average by coating	SD, μm	var. coeff., %
REFERENCE MATERIAL (precipitation hardened)	R1a_v30_f20_ SiC_t180_v1	30	20	843	885	31.6	3.6	908.2	34.2	3.8
				907						
				911						
				878						
	R1a_v30_f20_ SiC_t180_v2	30	20	865	902	28.4	3.1			
				902						
				933						
				909						
	R2a_v30_f20_ SiC_t180_v1	30	20	919	938	23.0	2.5			
				917						
				952						
				962						
FORMERA coating	F1a_v30_f20_ SiC_t60_v1	30	20	405	411	28.5	6.9	663.5	195.1	29.4
				390						
				453						
				396						
	F3a_v30_f20_ SiC_t180_v1	30	20	749	731	47.6	6.5			
				672						
				785						
				720						
	F1a_v30_f20_ SiC_t180_v5	30	20	848	848	8.5	1.0			
				839						
				859						
				845						
LUMENA coating	L3a_v30_f20_ SiC_t180_v1	30	20	759	754	14.4	1.9	743.7	52.0	7.0
				740						
				744						
				772						
	L2a_v30_f20_ SiC_t180_v1	30	20	792	798	4.6	0.6			
				802						
				800						
				797						
	L2a_v30_f20_ SiC_t180_v2	30	20	690	679	7.4	1.1			
				679						
				674						
				674						

5. GENERAL REVIEW AND EVALUATION OF THE INTEGRATED TEST RESULTS

An overview of the obtained numerical characteristics derived from these tests is provided in Table 20.

Summarizing the test results, we established that the hardness and the thickness of the Formera coating (1800 HV and 5.77 μm) was $\sim 20\%$ higher than that of the Lumena.

The instrumented scratch tests with progressive and constant load showed that Formera coating has better scratch resistance, i.e. it has 10% higher critical load ($F_{\text{crit}} = 115 \text{ N}$), and less sensitivity to gross spallation of the coating.

Ball on disc testing method was used to characterize the wear performance of the investigated materials. While we measured a higher friction coefficient of the Formera coating, as compared to Lumena coating, the wear damage estimated from the width of the wear track was significantly lower for the Formera coating. This can be explained by the much higher hardness and thickness of Formera coating, producing more abrasive wear debris, but better tribological loadability.

In conclusion, Formera showed better tribological performance than Lumena in terms of both scratch and wear resistance, because it is harder and thicker.

During this research work we had no possibility to make a comparison between identical the thickness of coatings, because we got the test samples from the industry as ready. Next time, we will make sure that the coatings on the samples are of identical thickness.

Altogether, the executed tribological, mechanical and morphological analysis demonstrate the importance, as well as justify the need for the multiple characterisation of the tribological behaviour of the wear resistant coatings.

Table 20. Summarizing table for all of the test results

Coating	Hardness test	Coating thickness (Calotest)	Prog-ressive load scratch test	Scratch width from constant load scratch test		Friction coefficient (μ) and wear track width (t) from ball-on-disc test	
	HV1	s, μm	F_{crit} , N	$W_F=50$, μm	$W_F=80$, μm	μ , –	t, μm
Uncoated	532	not relevant	no data			0.57	908
Formera	1863	5.77	115	94	124	0.55	664
Lumena	1529	4.80	107	84	120	0.44	744

6. SUMMARY

The objective of the research work was to compare the tribological behaviour of two industrially produced and applied advanced ceramic (CrAlN and TiAlN) coatings used for improving the lifetime of different cold work tool steels.

We have determined and individually examined specimens with different surface preparations. There were eight samples for each coating and also for the uncoated reference material.

The heat treatment process for these samples was accomplished in 3 stages i.e. heating, quenching and tempering, providing acceptable strength and hardness of the base tool material.

The surface preparation of the samples was done by polishing. It was followed by the coating process which is related to one of PVD based processes, called arc evaporation.

The analysis of hardness, calotesting, scratch testing and wear characterization was done on the samples of these two types of coating systematically, to evaluate the scratching and wear performance and to establish novel results.

Microscopic examinations of the scratch and wear track morphology of the samples were also performed, so as to provide a better understanding of material behaviour and to formulate more evident hypotheses relating to the possible microstructural reason for the obtained numerical results.

Scratch testing was done to characterize the adhesion of the coating and to evaluate the resistance of the surface to damage caused by a sharp indenter. The results were studied and the curves of the friction coefficient vs. scratching distance, as well as normal force vs. scratching distance, were created.

The amount of wear on any component has a great influence on its lifetime and maintenance or repair costs. Wear is thus a characteristic parameter in the entire tribosystem, whose analysis forms the basis of various theories and practical applications.

Ball on disc testing method was used to characterize the wear performance of the investigated materials. The experiments were performed on various specimens and with the help of data collected by the test machine the curve of friction coefficient vs. sliding distance were compiled.

The complex mechanical and tribological tests – involving, hardness test, ball cratering test, instrumented scratch tests and ball-on-disc wear test - showed that the Formera coating, which possesses higher thickness and hardness, and has more

favourable tribological behaviour, i.e. higher resistance against both scratching and ball-on-disc type wearing.

These findings are in good agreement with the intended uses of the coatings, presented on the manufacturer's website. FORMERA coating is mainly used on the surface of tools used for machining AHSS steels, while LUMENA coating, which has a slightly lower tribological performance, is applied on the surface of plastic machining tools, which are expected to be subjected to slightly lower loads.

In order to answer the contradictions and questions revealed during the research work, we will continue the examination of the coatings based on a larger number of samples and a higher number of measurement in the framework of our future professional work.

7. APPLICATION POTENTIAL OF THE RESULTS OF THE RESEARCH WORK

The tested coatings are already widely used in industrial practice, for which, however, few test results are available.

Considering that the complex characterization of tribological behavior presented in the current work is of novelty value, therefore hopefully, the results can be useful for the manufacturing company, above all for the further development of the technology.

As the coatings, we studied, were produced by applying the PVD arc evaporation coating technology, which is not widely spread in Hungary and international practice yet, the presented results may help the widespread use of these coatings, as well.

8. FURTHER DIRECTION OF RESEARCH WORK

The further direction of research work will involve multiple objectives:

- to compare the tribological behaviour of two industrially applied advanced ceramic coatings, as well as their duplex treated alternatives used to increase the wear resistance and lifetime of tools applied in metal forming of AHSS steels.
- to analyse the physical-microstructural background of the improved performance of the duplex treated surface coatings, as well as to study the influence of different processing methods on the structure and properties with the aim of technology optimization of duplex treatments with special emphasis on the tribological performance of these coatings.

To achieve these goals a complex tribological characterization of the investigated coating systems is suggested. The research focus is put on the development of an optimal technology combining nitriding and a subsequent PVD coating, i.e. optimizing their combined effect, which represents a relatively new research area.

The related task supposes not only producing surface layer with excellent properties for tribological applications but also providing an optimal “co-operative material system” composed of the differently produced layers. The final objective of the research work will be the technology optimization of a given combination of the substrate/coating/technology system, furthermore the quantitative and qualitative analysis of the tribological performance and failure process of the investigated mono- and multilayer coating systems.

- Test materials: Substrate: cold forming tool steel; Coating: hard ceramic layer materials (TiAlN, CrAlN based compounds)
- Applied heat treatments: nitriding + PACVD coating.
- Applied testing methods: ball-on-disc tribological test, hardness measurement, instrumented scratch test, ball-cratering method, profilometry, surface topography, microstructural characterization and morphological analysis of the wear tracks and scratch traces by optical and scanning electron microscopes, XRD phase analysis, FIBSEM 3D structural analyses of the coating layers.

As it was in the case of the reported research work, we are expecting the valuable support of our industrial partner, i.e. Oerlikon Balzers Coating Austria GmbH Magyarországi Fióktelepe (Balzers GmbH Mft.), Székesfehérvár, Hungary, in the future, as well.

The scientific novelty of the research area and the planned complex research program will provide an excellent possibility to get new scientific achievements, to prepare high-level professional publications on the related fields, and presumes on the attention of both the theoretical and practical engineers, furthermore industrial specialists.

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APPENDICES

Appendix 1.: Optical microscopic panorama photos of scratches taken by progressive loading

1.1. OM photos for FORMERA coating

1.2. OM photos for LUMENA coating

Appendix 2.: Friction coefficient/loading force vs. scratch length diagrams for progressive loading scratch tests

Appendix 3.: Friction coefficient/loading force vs. scratch length diagrams for constant loading scratch tests

3.1. Trial tests for LUMENA $F_{(min \div max)} = 2 \div 100$ N; $F_{grad} = 10$ N/mm; $L = 10$ mm

3.2. Final tests for FORMERA $F = 50$, and $F=80$ N; $L = 6.6$ mm

3.3. Final tests for FORMERA $F = 50$, and $F=80$ N; $L = 6.6$ mm

3.4. The measured scratch width and friction coefficient values for FORMERA and LUMENA COATINGS

Appendix 4.: Friction coefficient/loading force vs. scratch length diagrams for constant loading scratch tests

4.1.: Trial tests for Lumena $F_{(min \div max)} = 2 \div 100$ N; $F_{grad} = 10$ N/mm; $L = 10$ mm

4.2.: Final tests for Formera $F = 50$, and $F=80$ N; $L = 6.6$ mm

4.3.: Final tests for Lumena $F = 50$, and $F=80$ N; $L = 6.6$ mm

4.4.: The measured scratch width for FORMERA and LUMENA COATINGS

Appendix 5.: Friction coefficient vs sliding distance diagrams for ball-on disc test $v=30$ mm/s, $N=95.542$ 1/min, $t=180$ min, $F=20$ N