PART 2: PROGRAMME PROJECT INFORMATION

2.1. Project No. 6

Title	***Processing of metal surfaces to lower friction and wear***	
Project leader's name, surname	Karlis Agris Gross	
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2.2. Tasks and deliverables

(List all tasks and deliverables that were planned for reporting period, list responsible partner organizations, give status, e.g. delivered/not delivered)

Target: *Develop a method and criteria for the optimization of metallic material properties to improve the surface treatment and coating for reduced friction and wear of friction pairs including interaction with metal surfaces and ice.*

Time frame for the core tasks is given in Annexes 6-A.

Task 1 of the 2nd period: To characterize the metal surface and determine the best testing methods.

Planned tasks: (1) to develop a polishing method for smaller samples, (2) to analyse the surface of the samples with scanning electron microscope (SEM), two types of optical microscopy, atomic force microscopy (AFM), and profilometer, (3) to compare analysis results of each method used, (4) to prepare scientific publication

Task 2 of the 2nd period: To modify the slip-measuring equipment for laboratory conditions, and to prepare a climate simulator for experiments at low temperatures.

Planned tasks: (1) to prepare a climate simulator for operation at temperatures ranging from 0°C to -10°C, (2) to adjust laboratory equipment in accordance with the climate simulator dimensions (3) to determine the measurement sensor location for obtaining data with greater relevance, (4) to find information on where improvements can be made to the installation and/or climate simulators, (5) to develop a method for measuring the slip under laboratory conditions (deliverables).

Task 3 of the 2nd period: To modify the metal surface, and to determine the slip dependence on the modifications made.

Planned tasks: (1) to prepare metal samples with different degrees of surface roughness, (2) to prepare metal samples with different degrees of surface hardness, (3) to determine the reliability of slip measurement results, (4) to determine how the sliding ability changes with temperature (5) to submit an abstract and participate in the European Materials Research Society conference.

The second phase of the project objectives have been fully completed:

1. Detailed analysis of metal surface samples, using various analytical methods, has been performed. Results concluded that AFM provided the best quantitative and qualitative information and allowed for the detection of scratches from 4nm in size. In contrast, optical microscopy was shown to be the fastest method to detect surface scratches (approx. 72% of scratches were detectable by AFM). The ranking order of methods used for scratch detection is improved in the following order: profilometer, SEM, optical microscopy, AFM.
2. The custom-built, energy efficient, microclimate chamber was fitted with a cooling module, and is now capable of providing an internal air temperature ranging between +0°C to -20°C, with an accuracy of ±2°C (the desired average operating temperature -10°C). A slip stand was built for testing the slip of metallic materials on ice (adapted for use in the climate simulator). The optimisation of the ice-track freezing, and the manufacturing process, provided the necessary information to determine the inclination of the plane.

In order to optimise the automatic polishing of samples, sample holders were made. Experiments were conducted to obtain a variety of surface roughness by using different coarseness of sandpaper. A method for a smooth and repeatable surface was developed. The first experiments were to determine the effect of scratches on the sliding surface. Certain samples and air temperatures have an effect on the sliding ability. The first experiments were conducted on hardened samples.

Nr.	Tasks	Deliverable	Responsible partner	Status
1	Develop a method for measuring slip under laboratory conditions	Report (method) 31.12.2015.	K.A. Gross, Biomaterials research laboratory, RTU	Delivered Annex- NN
2	Develop a method for determining the slip under real track conditions, in comparison with laboratory equipment	Report (method) 30.06.2017.	K.A. Gross, Biomaterials research laboratory, RTU	In beginning
3	Optimise metal surface for increased gliding on ice	Report 30.06.2017.	K.A. Gross, Biomaterials research laboratory, RTU	In progress
4	Determine the relationship of gliding between the metal surfaces and ice (report)	Report 30.09.2017.	K.A. Gross, Biomaterials research laboratory, RTU	In progress
5	A recommendation for a modification of metal surface to improve gliding in track conditions, application of the material	Report 30.12.2017.	K.A. Gross, Biomaterials research laboratory, RTU	In beginning

In case of non-fulfillment provide justification and describe further steps planned to achieve set targets and results

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The planned targets of the NRP IMATEH Project 6 „ *Processing of metal surfaces to lower friction and wear"* were fully achieved in the reporting period from 01.01.2015 till 31.12.2016. The planned tasks are completed and the main results obtained.

2.3. Description of gained scientific results

(Describe scientific results achieved during reporting period, give their scientific importance)

Tasks for Period 2	Main results
1. To characterize the metal surface and determine the best testing methods	***A scientific paper published in the journal***

In order to determine the most appropriate test method for precise metal surface characterisation, different methods of analysis were tested and compared. The methods tested were: optical microscopy, scanning electron microscopy, atomic force microscopy and profilometer. The different methods were all tested on a polished steel sample, seen in Figure 1.a). Digital images were taken of the exact same area of the surface under study with the exact same proportions, which were marked by nano-sized indents (see red points in Figure 1.b). From the resulting scanned images, two small areas were examined. The two examined areas, with a size of 50x50 µm, helped to determine the best method of qualitative and quantitative surface characterisation.

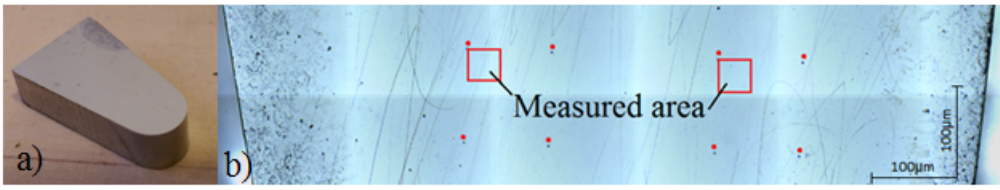

Figure 1: a) polished steel sample for analysis, b) analysed areas

Visual qualitative analysis of the data gave the resulting images (see. Fig. 2). The sharpness and level of detail in the images were evaluated and compared. In order to objectively compare each method, the resulting images were studied, visible scratches were counted and the percentage comparison between each method was calculated. Results showed that the image with the best resolution was given by the atomic force microscopy. With special software, quantitative analysis was done between the resulting images from the atomic force microscope and profilometer; this improved the ability to identify scratches on the profile cross-section.

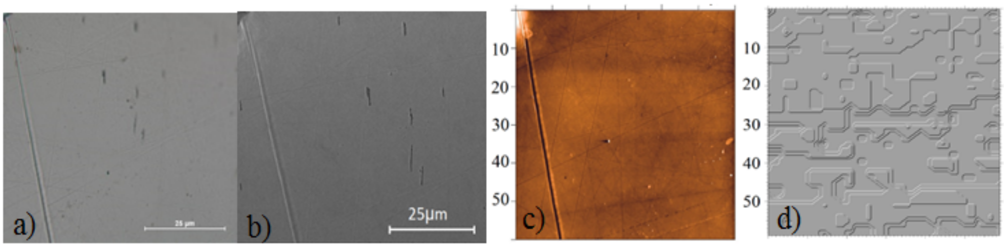

Figure 2: Different topography method results: a) optical microscope; b) scanning electron microscope; c) atomic microscope; d) profilometer

From results it was concluded:

1) Atomic force microscopy provides the best qualitative and quantitative indicators of the viewed surface, but it is expensive and has limitations for the size of the samples studied;

2) The success of scratch detection between different topography methods have been placed in the following order: profilometer, scanning electron microscopy, optical microscopy, atomic force microscopy;
3) Optical microscopy proved to be the fastest way to obtain a good quality image of the sample surface. Although much faster, the images provided about 70% of the information obtained by atomic force microscopy;
4) Atomic force microscopy allows up to 4 nm saddle scratches;
5) Profilometer method is not effective on such a small surface areas.

Scientific paper was prepared and published: Z.Butans, K.A.Gross, A.Gridnevs, E.Karzubova. “Road safety barriers, the need and influence on road traffic accidents.” Materials Science and Engineering (MSE), in 2nd International Conference on Innovative Materials, Structures and Technologies, S. Rucevskis and D. Bajare, Editors; 2015, pp. 1.-8.

2. To modify the slide measuring equipment for laboratory testing, and to prepare a climate simulator for low temperatures	***Equipment for measuring the sliding ability, which is suitable for a customised cooling chamber. Report (deliverable): "The method for measuring the slip in laboratory conditions"***

A device (slip stand), built to measure modified metal samples gliding on ice at a variation of experimental settings, has been developed. To complete the phase, tasks were carried out in the following order:

1. Design the stand concept and make stand prototype;
2. Modify climate simulator
3. Adjust the sliding bench for experiments in the climate simulation chamber;
4. Conduct pilot experiments.

Experiments using the slip stand evaluated the effects different surface treatments had on metal gliding on ice, and where appropriate, on other materials such as metal or plastic. The structure of the stand was based on the use of the inclined planes with optical sensors that measured the time the samples took to slip from the top to the bottom of the inclination. The time, in which the sample took to slip, was the chosen parameter to characterise the slip properties numerically.

During experiments the stand was located in the climate simulation chamber. Its design was easily customised for various geometry models, this flexibility in design opened up a wide range of experimental variations, to provide more detailed information.

The successful development of the ice track for laboratory experiments, was based on the duration of each test, refrigeration modes, ice treatment, and plane angle. With the developed methodology the required experimental conditions were achieved.

An extensive series of pilot experiments, helped to determine the various factors that influenced the results, i.e. sample weight, the temperature of the ice, the angle of the plane, and the absolute value of the dispersion. This helped confirm which variable most influenced the sliding of the metal on ice conditions under which the experiment is best observed in the surface modification effect. In consideration of the obtained experimental data, a methodology for future experiments was established.

The completion of the 2nd task of the 2nd phase saw the development of the slip measurement equipment, alterations to the climate simulator for laboratory experiments at low temperatures and the completed deliverable (report) “The method for measuring the slip in laboratory conditions”. The deliverable has been written in Latvian, 16 A4 pages long, it includes the following sections: (1) Sliding stand prototype, (2) Climate simulation chamber modifications, (3) Adjustments to the sliding stand for experiments in the climate simulation chamber (4) Pilot experiments in the climate simulation chamber (4.1) Preparation of the ice track for experiments, (4.2) To find the optimal inclination angle for the trace (4.3) The effect of temperature, in the climate chamber and of the studied sample, on the metal sample and ice quality.

3. Modify the metal surface to determine the slip dependence of the modifications	***Prepared report, submitted abstract, and participation in the European Materials Research Society Conference***

In consideration of the possibilities and limitations of the manufactured slip stand, 42 identical metal samples were made. These samples were polished to a mirrored surface using an automated polishing machine, for laboratory testing.

In order to obtain metal samples with different degrees of surface roughness, they were scratched with sandpaper. Since the manufacturer range of sandpaper is so large, sandpaper samples of different brands were compared, each test ensuring the same compression weight, number of scratches and scratch length. The developed samples were examined and compared under a microscope, and with 3D micro-topography measurements. The results found the best sandpaper brand suited for the samples. The deciding factor of the sandpaper was to ensure that all samples had a surface texture as similar as possible.

To improve reliability of the data collected from experiments, each experiment was repeated multiple times. Samples with different degrees of surface roughness were compared by timing the slip speed. These experiments were repeated over several days. Data was obtained on the number of required samples, how many times the experiments must be repeated, and the number of experimental days, to be sure that the experimental results were credible.

To determine the temperature and conditions needed for the experiments and the influence temperature had on the slip, the experiments were carried out at different temperatures. The results helped determine the optimal temperature to minimise systematic error, under the assumption good ice quality would not materially affect the sample roughness. A chart representing the sliding times, of differently processed samples, with a dependence on temperature, can be found in Figure 3.

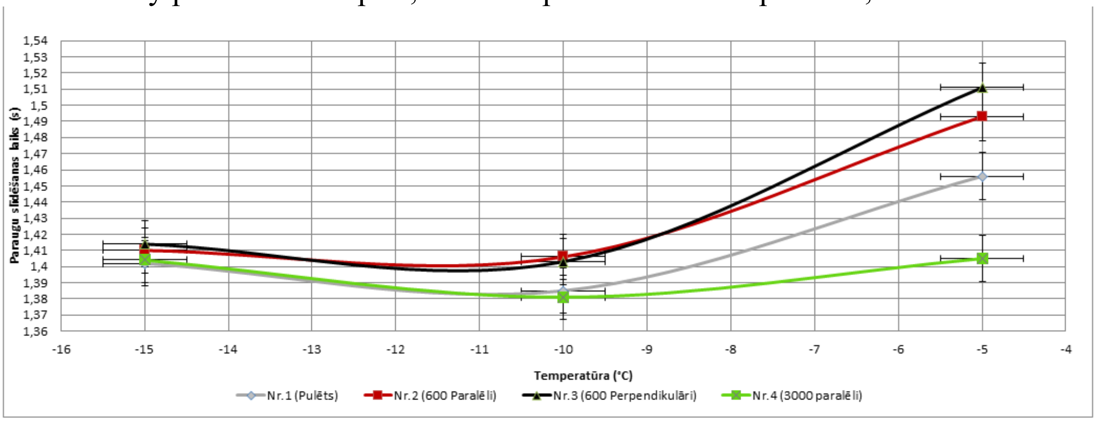

Figure 3. Sample sliding time with a dependence on the temperature

Experiments were conducted to obtain information about the effect of applied weight on the sample sliding time, with the aim to determine the best weight for the samples. The optimum conditions for the surface roughness, to be the only influencing factor in the experiments, were then chosen.

A method to measure the hardness of stainless steel was developed. After detailed research on the materials used, the experimental samples were heat-treated using an annealing furnace. The heat treatment process involves three stages: annealing, hardening and tempering. During the heat treatment process the prototype had formed an oxide layer, which was partially rubbed away to prepare the sample surface with a roughness of Ra≤2 μm. This was important to collect the correct hardness measurements. Initial results showed that the material hardness increased by ~30%.

An abstract was written for the participation at the European Material Research Society (EMRS) Conference "EMSR Fall meeting 2015", Warsaw, Poland, 15.09.2015-18.09.2015 with the poster: "Finding the best qualitative and quantitative assessment method for highly polished low-friction surfaces" (J.Lungevics. J.Zavickis, L.Pluduma, K.A.Gross). The best and most effective method to polish metallic surfaces for qualitative and quantitative evaluation was presented at the conference. The greatest benefit of this study is the idea of how effective and useful it is to have a variety of surface evaluation methods that, with further progress of the project, will allow rational decisions on optimal selection of equipment. Multiple ideas, from other research reports, on a variety of surface modification methods to improve thermal resilience, and slip and abrasion resistance properties have influenced the methods chosen for this project. This information allowed for future research in the direction of the project and a way to achieve the objective pursued.

2.4. Further research and practical exploitation of the results

(Describe further research activities that are planned, describe possibilities to practically exploit results)

With the use of the slip measuring equipment (the laboratory track) and the development and customisation of the climate chamber, the first two phases of the project were fully completed. Great care was taken to ensure precise methodology for the assessment of various parameters and their influence on the measurement results. As a result, measurement repeatability and reliability of results was achieved. A study of various metal surface analysis methods helped to discover the advantages and disadvantages of each method, this allowed for the selection of the most appropriate test method.

The results achieved and developed methods are the basis for all subsequent experiments, to evaluate the influence of a variety of metal surfaces on gliding.

During the project phase 3 a research on different methods for metal surface modification will be continued:

- Roughness - the first experiments were conducted in phase 2, with plans to evaluate the effects of surface roughness on sliding, to determine the best degree of surface roughness and texture.
- Hardness – research in the second phase showed that the modified samples have shown a slightly better sliding time than untempered sample. In order to judge the full impact thermal treatment has on the gliding of the samples, there will be more in-depth studies using statistically reliable measurements, such as a larger number of samples. The comparison of scratching resistance between tempered and untempered samples, and the examination of how different temperatures effect the slip properties of these samples will also help to judge the impact thermal treatment has on sliding speed.
- Chemical modification - The metal sample's surface hydrophobia will be measured and modified to improve its relation to gliding.

Work on the development of surface modification methods for the large metal samples has also begun.

Tasks for the 3rd period:

1. Modify the metal surface, to determine the slip dependence of the modifications
   - Expected results: Discover the best direction for surface modification, which will increase metal on ice sliding properties.
   - Participation in the conference and publication.
2. Develop methods for improving the gliding surface of a larger surface under real conditions

- Expected results: Data collection, compilation, and analysis. The beginning of new methods for larger metal surface modification.

One of the major issues during the execution of the second phase of the project was due to personal changes in the project staff, this occurred mainly due to limited finances. Lead researcher, J.Zavickis, stopped work on the project due to changes in employment, as a result, the project manager had to take over his duties and become more actively involved in the planning of experiments.

Limited project funding in phase 3 could also cause changes in staff, but all project employees are actively involved in all the planning of experiments and discussions, as well as ensuring all experimental results were uploaded to the Dropbox folder, to minimize the impact of staff turnover during the project.

2.5. Dissemination and outreach activities

(Describe activities that were performed during reporting period to disseminate project results)

Submitted conference thesis:

1. J.Lungevics. J.Zavickis, L.Pluduma, K.A.Gross. "Finding the best qualitative and quantitative assessment method for highly polished low-friction surfaces". EMSR Fall meeting 2015", Warsaw, Poland, 15.-18.09.2015.

Conference participation (poster presentations can be viewed in the project website):

1. '*European Material Research Society (EMRS)*' conference "EMRS Fall meeting 2015", Warsaw, Poland, 15.-18.09.2015, with the poster: "Finding the best qualitative and quantitative assessment method for highly polished low-friction surfaces" (J.Lungevics. J.Zavickis, L.Pluduma, K.A.Gross).
2. International conference "Innovative Materials, Structures and Technologies" (IMST 2015), Riga, Latvia, 30.09. - 02.10, with the poster: "Road safety barriers, the need and influence on road traffic accidents" (Z.Butāns, K.A.Gross, A.Gridnevs, E.Karzubova).

Participation in scientific seminars:

1. "Meeting of s-SNOM practioners", Garmisch, Germany, 09.06.15 - 11.06.15 Key findings: The seminar brought together users of optical microscopy to characterize materials. There is great interest to combine results from the chemical and structural analysis, to provide a much greater insight into the material surface. Workshop participants examined the capability of infrared for the analysis of various depths, and by using atomic force microscope tip, were able to explore the topography at nano-size. It was presented in studies, that such methods are already being used to describe the material areas at the nano level, proving success of the method. After the meeting, the company Neaspec, presented the capabilities and possibilities of SNOM instruments, related to surface characterisation. The information gathered from the seminar showed the possibilities that would bring great benefit to the surface of material characterization.

Full-text scientific papers (can be viewed in the project website):

1. Butans Z., Gross K.A., Gridnevs A., Karzubova E. Road safety barriers, the need and influence on road traffic accidents, IOP Conference Series: Materials Science and Engineering Volume 96, 2015

a. http://iopscience.iop.org.resursi.rtu.lv/article/10.1088/1757-899X/96/1/012063/pdf

Bachelor thesis in progress (scheduled to be defended in June 2017):

1. Klavs Stiprais, "Metal treatment to reduce friction", overseen by Asoc. Prof. K.A.Gross.

Master's thesis in progress (scheduled to be defended in June 2016):

1. Jānis Lungevičs, "Friction and wear-reducing surface treatment methods for the assessment of tribology properties", overseen by Prof. J.Rudzitis.
2. Ernests Jansons, "The effects of surface roughness on the sliding of metal on ice couples", overseen by Prof. J.Rudzītis.

Doctoral thesis in progress (scheduled to be defended in 2017):

1. Žans Butāns, "The mechanical properties of road safety barriers surface and its impact on traffic, accidents, and safety of passengers", overseen by A/Prof K.A.Gross

Completed project deliverables (written report):

1. The method for measuring the slip under laboratory conditions

Dissemination of results:

Within the second phase of the project two project meetings (17.04.2015. and 14.10.2015.) were organised. Researchers, staff and industry representatives attended and discussed the project tasks and deliverables. Internal meetings are regularly (at least once a month) held to discuss work progress, with the attendance of the project manager and project staff who are directly involved.

The IMATEH website (http://imateh.rtu.lv/) provides information on the activities of the project, publications, conferences and current events.

Leader of the project No. 6 ______________________ Karlis Agris Gross____________________

(signature and transcript)

(date)