Materials Fast Track - Sharing our learnings

- An overview of projects from Materials Fast Track

fast track

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Introduction

With the aim of increasing the competitiveness of Danish industry

Materials Fast Track is a consortium consisting of Siemens Gamesa Renewable Energy, Hempel, Elplatek, Terma, FORCE Technology, Danish Technological Institute, Aalborg University and Technical University of Denmark, which has created a societal partnership with the aim of increasing the competitiveness of the Danish industry and creating new, as well as maintaining existing, jobs in Denmark through implementation of the right materials solutions. The Fast Track societal partnership was launched at the beginning of 2016 through support by Innovation Fund Denmark (IFD).

The beginning of a new community

At the beginning of the project period, Materials Fast Track was mainly focusing on bringing together experts from the project partners to solve the challenges met within the Danish industry in a new and agile project model. This problem-solving model was named the Fast Track Expert Panel and was a new and fast way to accelerate the implementation of new knowledge and an agile way of implementing new material solutions based on the best ideas from universities, institutions and companies. Clearly, it was the vision to facilitate rapid access for the Danish industry to world-leading, advanced materials technology, nationally as well as internationally.

Since then, further to establishing and optimising our Fast Track Expert Panel service, it has been possible to add further services that facilitate knowledge sharing between universities, institutions and companies into what we call the Materials Fast Track Community, which covers what we call the Materials Fast Track Specialist Network. A truly trust-based confidential forum for knowledge and idea sharing, problem-solving and cross-industry networking among materials specialists.

Figure 1: The Materials Fast Track Community schematized with a feedback loop where it is the idea that a material challenge arises within the network and is evaluated by the expert panel who, together with the challenge owner, discusses the challenge. Either the discussion is ended by a quick standardised commercial solution or it can be decided to move on into a Fast Track project in collaboration with the Fast Track expert partners. The knowledge gained from the Fast Track project can evolve into additional funding opportunities in e.g. Horizon2020, Innovation Foundation, Innobooster, etc. The specialist network could easily enable new relations and potential partners for an additional funded project beyond the involvement of Fast Tracks.

The Fast Track Materials Community is a concept that enables the use of the Expert Panel services through the trust-based relationships created within the Specialist Network, to feed additional material issues into the Fast Track Expert Panel. The Expert Panel will subsequently evaluate the challenge together with the problem owner(s) and decide if the challenge is solved by discussing it with the Fast Track Expert Panel or if a Fast Track Project should be initiated. The learnings generated by the Fast Track projects will be presented in a network meeting to spread the knowledge generated.

What our Fast Track Expert Panel does

Our **Fast Track Expert Panel** specialises in helping innovative companies, guiding problem owners to the right solution solving the material challenge; whether it is an unexpected corrosion problem, materials fatigue, finding the proper surface treatment or guidance for material selection.
The Fast Track Expert Panel offers:

- **Direct access to the right experts** who can give the RIGHT solution.
- **Free advice and guidance** from the expert panel.
- Possibility of rapidly starting up a project with the **right partners as participants to the get the right solution** – and not just A solution.

The Fast Track expert panel consists of leading materials experts from FORCE, DTI, DTU, Aalborg University, Siemens Gamesa Renewable Energy and Elplatek. Currently the expert panel is being expanded with international experts to ensure that the best experts will always be available within the panel. We believe that we have assembled the strongest group of materials experts, with a vast experience ready to solve incoming challenges aiming at acting with agility and confidentiality towards the customers.

The expertise in the expert panel currently covers the materials-related areas stated in the overview below.

Metallurgi	Electroplating	Paint	Tribology
Polymers	GFR Polymers	Materials modelling	Sintering / Casting
Ceramics	Aluminium	Corrosion	Additive Manufacturing
Welding	Characterization	Surface treatment	Advanced Manufacturing

Fast Track Specialist Network – a catalyst for creating your professional network

In our Materials Specialist network we aim to improve each other by facilitating a **cross company specialist network** within materials science and technology, establishing trust between materials specialists across the Danish industry.

We aim at **having 4-6 annual network meetings** in each network group, hosted by the group members. The host presents the company and either the daily materials challenges or specific project challenges met by the host. The groups are presented to different relevant topics, either by internal group members or by external experts bringing new and inspiring inputs.

Materials Fast Track is the facilitator of these meetings and provides a relevant agenda, sends out invitations and finds the host. The duration of these meetings varies between a half and a full day of networking and is rounded off with a social event with a focus on creating an open atmosphere in which ideas and daily challenges are discussed in a closed and confidential forum.

8 GREAT REASONS TO JOIN A NETWORK GROUP

A network by specialists – for specialists

We have a strong technical background with close ties to the Danish industry

Strong network – between meetings too

Stay in touch between meetings for technical feedback or discussions

Careful selected groups

The network groups are created with focus on professional competence. No conflict of interest

Brain sharing

Sharing is the key. By gathering specialists within their fields in one room, ideas will arise and problems be solved

Relevant topics – guaranteed

The themes of each meeting are carefully selected as a collaboration between the network manager and members. The themes are fitted to be relevant for the group

You are your network

There is an increased focus on the value of your network. Make yourself more attractive – expand your network

Idea sharing outside your industry

By combining experts from different industries, innovation will occur

Start-up and big companies

By combining specialists from big companies with specialists in start-up firms, interesting partnerships may develop

Focus on solving challenges from the industry

Since the beginning of Materials Fast Track, the aim has always been to meet the needs of in the industry. With this mindset, two different definitions of projects were set up: Fast Tracks and R&D Tracks.

Fast Tracks aim at meeting the immediate needs, solving a given challenge within the shortest possible period. A problem-solving model has been set up to ensure that a challenge is acted upon as fast as possible, establishing the connection between the problem owner and the right group of experts.

R&D Tracks, on the other side, focus more on R&D and transfer of research knowledge from the universities into development and hereby into real products. All the R&D Tracks are defined by a Fast Track industry partner with another Fast Track industry, RTO and/or university partner involved.

Common for both types of projects is that we aim at sharing as much knowledge as possible generated by these projects. This is done by presenting our work during the specialist network days that we give during the year.

In the following sections, Fast Tracks and R&D Tracks are described in more detail.

What is a Fast Track?

Fast Tracks are projects focused on rapid development or problem solving with a small team. The aim of a Fast Track is to find a given solution to an industrial problem within a maximum of 6 months. Figure 2 shows two examples of Fast Tracks. The "Research, Development, Implementation" process bar shows the timeline of the project coupled to the increase of TRL (Technology Readiness Levels). Our aim is to ensure that real value is generated from the Fast Tracks. It is often difficult to measure value generated directly from a project, instead a measure of increased TRL levels is used. The partner logos above the given Fast Track show the partners that have been involved in the project.

There are two types of Fast Track activities:

- **Internal Fast Tracks**: Fast Tracks initiated by a project partner. The budget for the internal Fast Track will be allocated via the societal partnership budget.
- **External Fast Tracks**: Fast Tracks initiated by external companies. A budget for the external Fast Track will be allocated by the consortium and the external company.

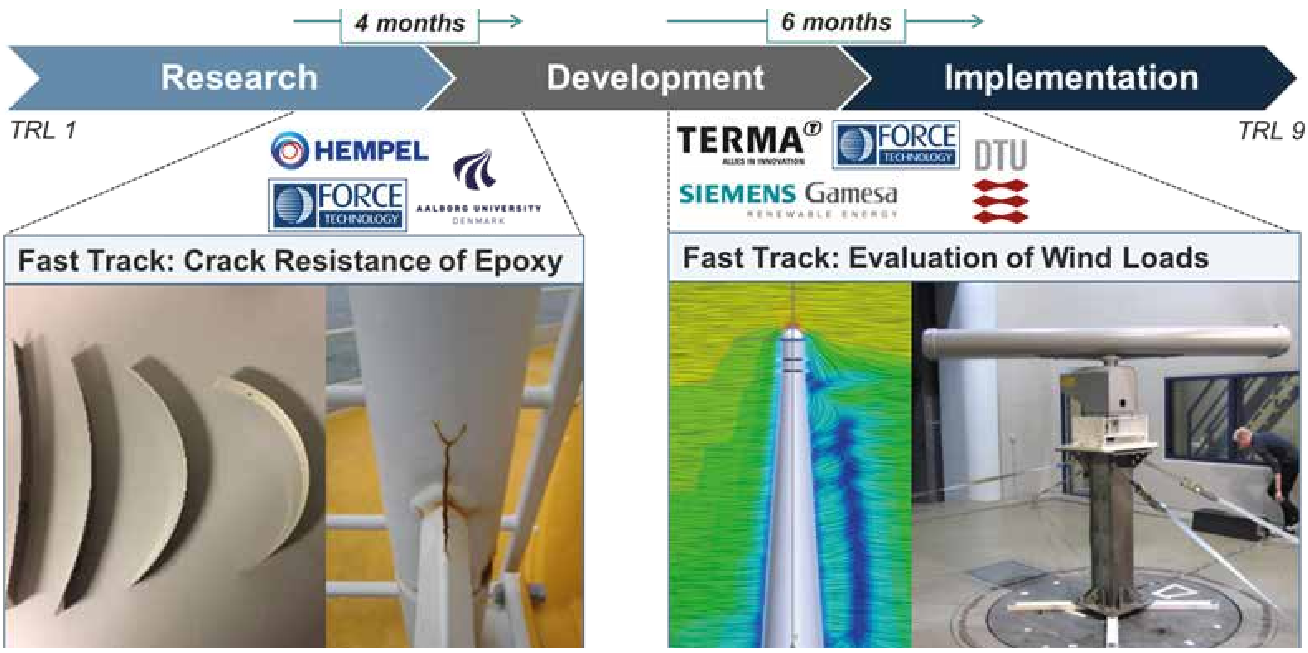


Figure 2: This figure shows two examples of previously conducted internal Fast Tracks. The "Research, Development, Implementation" process bar shows the timeline of the project coupled to the increase of TRL (Technology Readiness Levels).

Later in this brochure, you will get a thorough description of the Fast Tracks that we have been running throughout the lifetime of Materials Fast Track.

What is an R&D Track?

R&D Tracks are research and development with duration longer than 6 months aimed at solving a challenge identified by the industry. The R&D Tracks are initiated if they are "high impact solutions that can generate commercial value at the industry partners of more than 100 million DKK". An overview of the R&D Tracks can be seen in Figure 3. This figure also utilises the TRL scale as benchmark for the value creation, in this figure sketched as an example.

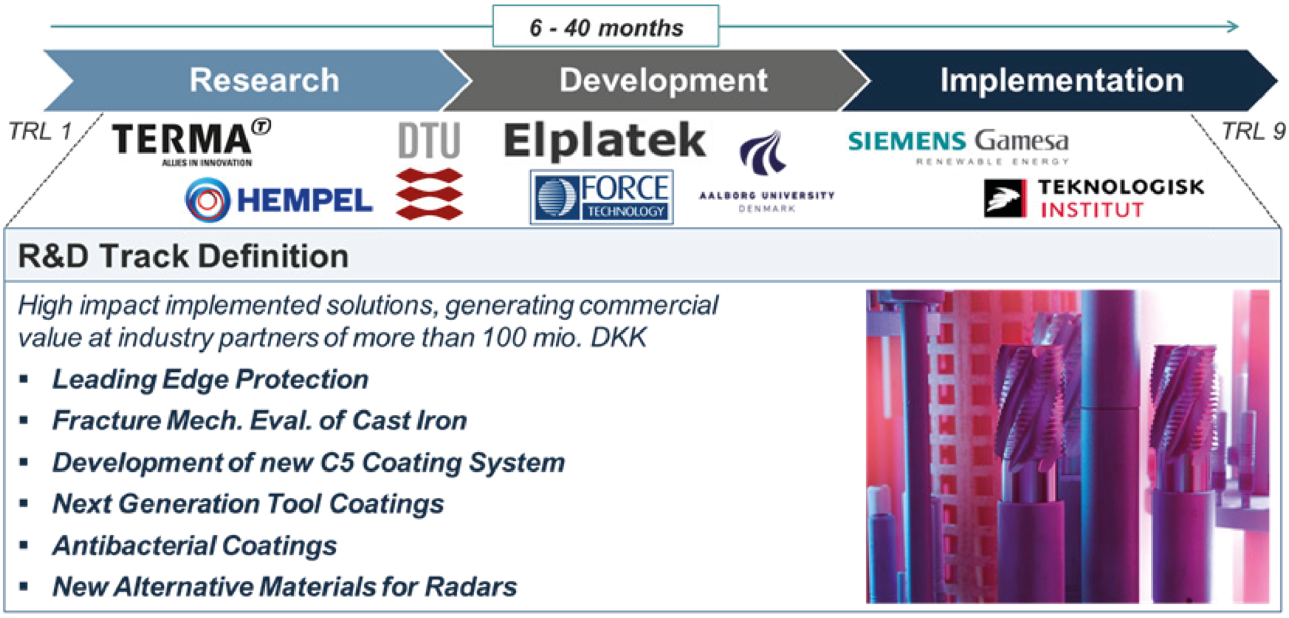


Figure 3: This figure shows an overview of the conducted R&D Tracks. The same idea of measuring the value creation in increase of TRL level is pictured.

The R&D Tracks in Materials Fast Track will be described in a later section of this brochure.

Why this brochure?

The aim of this brochure is to provide information to all who are interested and open to get inspiration from the work done in the Materials Fast Track project. We have tried to give an overview of all the technical projects, focusing on giving a brief overview of the purpose and the work conducted in the projects. Furthermore, we have added contact information to the respective Project Managers to enable you to contact them directly if you need further information or perhaps have new ideas on how to take the project even further.

Besides giving an overview of the projects, we have tried to sketch the value created in the projects. This is not always easy, especially when you focus on R&D Projects. We hope that sharing as much technical information as possible, both in this brochure and in the presentations, will enable creation of even further value for you and facilitate implementation of the learnings.

We hope that you will enjoy reading the brochure and are able to gain new knowledge and inspiration.

RD Platform 1: Characterization and failure analysis

SUMMARY

The service life of advanced machinery working in remote locations e.g. wind turbines, radars and ships are determined by the durability of critical components like blades, gears, bearings, fasteners, electrical connectors and paint systems. The frequency of maintenance and repair must be reduced and be planable to avoid costly shutdowns. Further improvements may be obtained by using new advanced materials solutions, enabling lower stress concentrations, more resilient lubrication, improved welding procedures, long-term corrosion and erosion protection, or better utilization of the material properties. Understanding of failures is a key enabler in overcoming these challenges.

This platform has developed a common "best practice" road map for failure analysis of critical components. The road map is initially based on state-of-the-art, literature and partners' experience in order to apply this in the Fast Tracks. The overall goal is to quickly understand and define the key problem to be solved taking the following factors into account: the full matrix of component, material, design, application and environment, as well as evaluating possible solutions taking this matrix into account.

Throughout the entire Fast Track project period the platform has acted as a supportive function for the R&D Tracks and Fast Tracks. Furthermore, we have participated and presented the platform knowledge at several events. Worth mentioning is the manuscript submitted and presented at NACE 2018 Corrosion conference in Phoenix, US in April 2018. The focus of the paper is to underline the necessity of cross-company collaboration, which were illustrated by presenting several cross-company projects conducted in Fast Track. Specifically, the presentation took its basis in optimization of corrosion protection of future offshore wind structures, highlighting the work flow, work methods and the hitherto obtained experiences from the various projects.

RESULTS & DISCUSSION

Failure analysis - definition

From [1] "failure analysis" is defined as:
"The logical, systematic examination of an item or its diagrams to identify and analyze the probability, causes, and consequences of potential and real failures".

The failure analysis can either be a physical investigation of a failed item, or it can be an inductive reasoning to identify potential failures, used as a preventive action or during prototyping before a product is marketed. The ultimate failure analysis is a root cause analysis, which covers all possible factors, which may have led to the failure. A physical failure analysis is the simpler investigation, which search for the failure mechanism of the physical component.

All the above-mentioned levels of failure analysis will be described in the following; with the main focus being on physical failure analysis.

Preventive failure analysis

Engineers have always had to consider the effects of a component failure on the systems and structures that they design. However, formal methodologies for these types of analyses were not developed until the early 1960s when the obvious safety and reliability requirements of the aerospace industry began to demand them. In the late 1960s several professional societies began to publish procedures for performing a Failure Modes and Effects Analysis (FMEA).

The analyses are intended for use by organizations as a tool for assessing the safety and reliability of system elements, or as a part of their product improvement processes. The latter includes analysis throughout the design process, from early in the conceptual stage to implementation and production [1].

FMEA and FMECA

Failure mode and effects analysis (FMEA) is a procedure by which each potential failure mode or fault of a system is analysed to determine the consequences or effects thereof on the system. This is done to classify each potential failure mode according to its severity, and to recommend actions to eliminate, or compensate for, unacceptable effects.

Failure mode, effects and critically analysis (FMECA) is an extension of the FMEA procedure to include assessment of the failure mode severity and probability of occurrence.

FMEA and FMECA activities help identify potential failure modes based on experience with similar products and processes—or based on common physics of failure logic. It is widely used in development and manufacturing industries in various phases of the product development.

Root cause analysis

A root cause analysis is a method used to identify root causes of failures or problems. A root cause is the ultimate cause or causes that, if absent, would have prevented occurrence of the failure. In [2] a root cause analysis is defined as "the combined action of three lower level failure causes; physical, human and latent failure causes". The term "physical" covers the physical mechanism, under which the item failed. Examples are fatigue, overload or corrosion. The human factor is, as the word indicates, the human factors, which can play a role in a failure cause. Finally, "latent" covers the indirect factors such as cultural or organizational rules that may lead to the human causes. That could be lack of inspections or insufficient education of the staff. When all three factors are considered, the failure analysis are considered to be a root cause analysis.

It is essential that a root cause analysis is performed systematically and with greatest effort, in order to reduce the risk of missing true root causes identification. The depth of the analysis is also an important parameter. It can be a simple 1-hour analysis and up to several months, if necessary. It depends on the severity of the problem and the effect of it also. There are many approaches in literature to a systematic root cause analysis. In the following, a few of the present methodologies will be summarized.

It should be mentioned that sometimes the term "root cause analysis" is incorrectly used when a simple physical failure cause analysis is in question. A true root cause analysis is the ultimate analysis of all possible factors, which may have played a role in causing the failure.

Fishbone diagram

Fishbone diagrams are named after their shape, which looks like a fish skeleton. They are also known as Ishikawa diagrams, named after the inventor [3i]. The diagrams are used to break down root causes that potentially contribute to a particular event, such as a failure. The causes are identified through brainstorming sessions, by going through a number of categories which may contribute to the overall event. The categories can differ from organization to organization, but a traditional categorization is as follows; people, methods, machines, materials, measurements and environment. For each of these categories, the possible causes to the present event are identified. For each of these identified causes, the question "why?" is asked again, and any answers are incorporated as smaller bones coming off that bone. For a complex failure, the fish can have many levels of bones.

Below in Figure 1 is shown a fishbone diagram, representing a problem with a car that will not start. As seen from the diagram, the potential causes are identified, within the six categories.

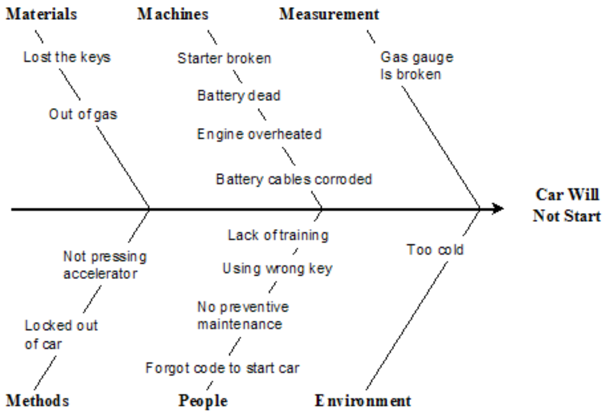


Figure 1. Example of how to use the fishbone diagram, in this case with a car that would not start [3].

5-why's

As the name indicates, the 5-why's method applies asking the question "why" repeatedly, which in the end may lead to explain the root cause of a problem. The number 5 is optional; it can be asked as many times as it makes sense, however, 5 is a reasonable number. The 5 why's method is a quick and simple approach to an understanding of a problem; and it is used as an individual analysis or a part of a deeper root cause analysis, for instance in combination with the fishbone method [4].

A3 problem solving

A3 is simple and strict approach to problem solving and continuous improvement, applying a template of the size of an A3 paper sheet, see Figure 2 [5]. The method is employing tools from other methods, such as an Ishikawa diagram (not mentioned here), the "5 Why's" method and another principle called Plan-Do-Check-Act referring to the various actions in the workflow. A3 problem solving follows a number of steps, which may vary, but most often the following 8 steps are included:

- Problem identification (PLAN)
- Problem clarification, e.g. "5 Why's" (PLAN)
- Setting target (PLAN)
- Containment (PLAN)
- Cause and effect, e.g. Ishikawa (PLAN)
- Follow up action (DO)
- Check results (CHECK)
- Share successfully implemented actions (ACT).

All the steps are reported in the worksheet and followed up upon. It provides a clear and concise method of reporting information, and allows for continuous improvement, as the process is visible and accessible to all. The method is typically used by lean manufacturing practitioners [5].

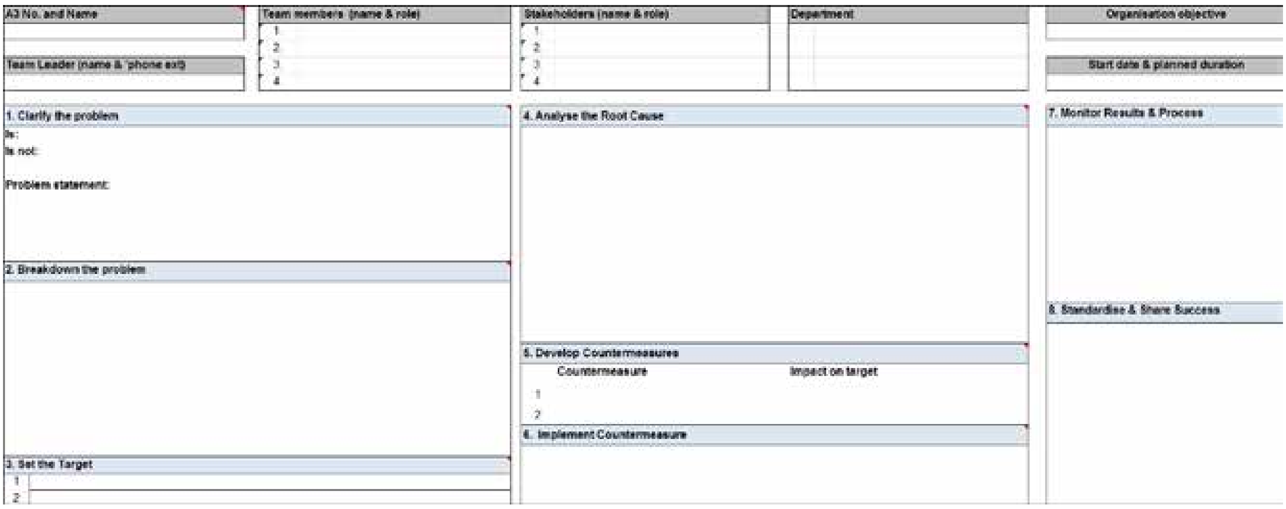


Figure 2. Example of the worksheet used in "A3 problem solving", which has the size of an A3 sheet [5].

Physical failure analysis

Until now the descriptions have treated the methodologies used to perform the all-embracing root cause failure analysis. It has become clear that many approaches exist to establish the overall failure cause. On the other hand, if a physical component has failed, a physical failure analysis is also needed, in order to determine the physical failure mechanism. This is referred to as the physical failure analysis. It covers a determination of the actual physical failure mechanism, such as corrosion, fatigue, overload etc.

A physical failure analysis can be described more specifically by a route of actions and investigations in the laboratory, as will be done in the following. In this study the focus is on a metallurgical failure analysis covering mainly metallic samples. If polymers, ceramics or other types of materials are in question, more investigations may be needed. But this will not be treated here.

Step 1: Identify problem

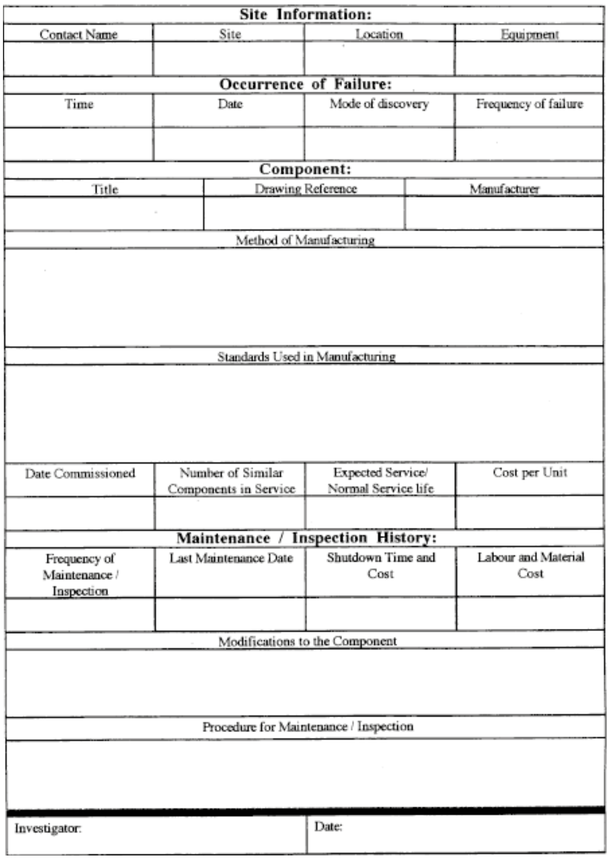
The initial part of the failure analysis is the most crucial and critical step, where most errors can be introduced. Imagine you receive a failed component; what to do?

First, there is a visual examination of the received parts. Perform as many observations, measurements and documentation hereof as possible, before doing any destructive impacts. If the failure is located on one side of a plate, try also to look at the other side. There might be important evidences hidden in areas far away from the obvious failure.

Then the failure must be confined. What is the primary failure and what are secondary failures? This is a really good question and may not necessarily be answered in this initial process. Representative samples are selected for further investigation. If the failure cannot be confined easily, more samples are necessary to include in the analysis.

The selected samples may have to be removed from larger parts. Make sure to preserve and protect surface to maintain important features. Be careful not to introduce any heat impacts when cutting or demounting parts.

Now samples are ready for the next step; analysis. But before performing any analyses in the laboratory, the other essential part of Step 1 is to get an understanding of the conditions, under which the part was operating. Therefore, data and background information must be collected. A simple questionnaire is a good place to start, see figure 3. Unfortunately, in many instances the investigator will receive only very little information about the history and operating conditions of the failed part. Therefore, an existing knowledge on components is a prerequisite of the investigator. Alternatively, assisting tools to help with this. This is described in the section "Necessary prerequisites".



Site Information:			
Contact Name	Site	Location	Equipment
Occurrence of Failure:			
Time	Date	Mode of discovery	Frequency of failure
Component:			
Title	Drawing Reference	Manufacturer	
Method of Manufacturing			
Standards Used in Manufacturing			
Date Commissioned	Number of Similar Components in Service	Expected Service/ Normal Service life	Cost per Unit
Maintenance / Inspection History:			
Frequency of Maintenance / Inspection	Last Maintenance Date	Shutdown Time and Cost	Labour and Material Cost
Modifications to the Component			
Procedure for Maintenance / Inspection			
Investigator:	Date:		

Figure 3 Example of simple questionnaire used in physical failure analysis.

Step 2: Analyse

Next step in a failure analysis is to analyse the selected samples. This can be varied endlessly and there is no limit to how many analyses one can perform. The real challenge lies within limiting the number of analyses to perform only the ones really needed. If the answer comes after the visual examination, there is no need to continue. But then comes the question; is this evidence enough? This is only a question which can be answered by the experienced investigator. Therefore, you cannot implicitly follow the same route of investigations every time; it has to be fitted to the component. However, the tool box, from which you select the analysis methods, is always the same. The most common methods applied in a failure analysis include the following:

- Non-destructive testing
- Macroscopic examination
- Microscopic examination
- Metallographic examination of microsection
- Mechanical testing
- Fracture mechanical computation
- Chemical analysis
- Laboratory scale experiments to simulate and test real conditions
- Modeling and calculations.

One important action throughout a physical failure analysis is to identify primary failures from secondary failures. What has caused the initial failure of the component?

Step 3: Conclude

Once all the data is gathered, the investigator must draw a conclusion based on the obtained results and evidences present. This requires that the investigator draw heavily on background experience and research performed. This leads us to the next chapter, about necessary prerequisites.

Necessary prerequisites

An important prerequisite in failure analysis is to be well informed on how similar systems operate and how they usually fail. 30 years of experience with coal-fired boilers will give a person a certain knowledge on possible causes of a tube leakage on a coal-fired boiler, which enables a straight forward process of finding the failure cause. However, 30 years of experience is not what everyone holds and there are other methods to search for knowledge, which can support the investigator.

In the literature many collections of case stories of failure analyses already performed exist, and these can be browsed through for inspiration. Nowadays searching is performed digitally by entering a few keys words, but earlier they were published in printed books and collections, from where the investigator needed to spend some time searching and researching. It might take a while to gain enough information about the type of component and application that you are working on, in order to solve the question.

The analyst must be aware of the normal, or expected, location and type of fracture in any type of part since any deviation from the normal must have been caused by some factors that must be discovered. Many databases and collections exist, where statistical data and background information on failures are listed. For the oil and gas industry, a commercial failure database is available; OREDA. OREDA is a database which provides data on failure rates, failure mode distribution and repair times for equipment used in the offshore industry. Statistics benefits a failure investigation by inspiring the investigator with possible failure causes and the chances hereof.

Statistical data must be handled with care. What makes sense in one industry may not in another. Also, statistics may differ within different companies. It may be necessary to create your own statistics in the company, in order to gain the full benefit from it.

Constraints and possible errors in failure analyses

Many constraints may arise when conducting a full root cause analysis, and this will hinder a true conclusion. The following list of typical constraints are inspired by [4]:

- Incorrect or insufficient data, too many assumptions
- Incorrect problem definition
- Cost and schedule constraints

- Rushing to a conclusion, which may not be correct. When first conclusion is found, the investigator tends not to continue the search
- Lack of knowledge and insight.

Many errors can occur during a failure analysis, which may ultimately lead to the wrong conclusion. Below is listed a number of the most obvious errors in a failure analysis [4]:

- Incorrect team composition
- Incorrect data classification, too many assumptions
- Lack of objectivity (you think you know the conclusion from the beginning)
- Cost constraints
- Lack of management commitment
- Rush to judgement
- Lack of insight.

Best practice road map for physical failure analysis

For the experienced investigator, a physical failure analysis is an intuitive process based on years of experience. The process is not following a predetermined route of actions but is an ongoing process based on the gained inputs. However, if the investigator does not possess years of experience, there must be a best practice road map that one can follow. In many publications, recommendations are made on how to perform a physical failure analysis. In this study, inspiration has been achieved from the literature and also from interviews with various experienced failure analysis investigators. Based on this, a best practice road map is presented in Figure 4.

Identify problem

- Visual inspection of received samples
- Identify primary failure from secondary failures
- Select representative samples for investigation
- Collect data and background information

Analyse

- Non-destructive testing
- Macroscopic examination
- Microscopic examination
- Metallographic examination of microsection
- Mechanical testing
- Fracture mechanical computation
- Chemical analysis
- Laboratory scale experiments to simulate real conditions
- Other

Conclude

- Summarize results
- Correlate results with background information
- Determine failure mechanism
- Perform investigations to support conclusion, if needed

Figure 4. Best practice road map for a physical failure analysis of a metallurgical component

References

[1] SAE Aerospace, "Aerospace Recommended Practice ARP 5580," 2012.

[2] D. P. Dennies, How to Organize and Run a Failure Investigation, 1st ed. ASM International, 2005.

[3] "Fishbone diagram for failure analysis." [Online]. Available: https://www.spcforexcel.com/knowledge/root-cause-analysis/analyzing-cause-and-effect-diagrams.

[4] R. J. Duphily, "Root Cause Investigation Best Practices Guide," 2014.

[5] "A3 Problem Solving." [Online]. Available: https://en.wikipedia.org/wiki/A3_problem_solving.

VALUE / IMPACT

The platform has contributed with knowledge about failure analysis and characterization to both internal and external companies involved in Fast Track. It is difficult to specify the exact value of the outcome, when it comes to new employment, saved expenses or likely. However, we have intended to broaden the knowledge about the topics and presented the road-map for conducting a failure analysis. That enables other companies to get inspired and enabled to perform failure analyses more easily in the future.

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Duration of project:
January 2016 – December 2019

Collaborating companies:
FORCE Technology (leader), all partners

RD Platform 2:
Materials and surface solutions

SUMMARY

R&D Platform 2 deals with fundamental development of next generation innovative surface solutions based on electroplating, thermal spraying, thermochemical treatment, PVD and new paint formulation through three R&D tracks and several external Fast Tracks.

Detailed activities are reported in R&D Track C: Development of new C5 coating systems; R&D Track D: New chemically resistant paint systems and in R&D Track E: Next generation tool coatings for machining composite materials as well as in several external Fast Tracks involving external partners. This is especially #11 Surface treatments of towers, #15: Viable substitute for chrome-VI surfaces in food contact zones and optimization of surface tests.

RESULTS & DISCUSSION

As to the long-term development activities, the main effort has been on PVD, electroplating and different paint activities:

PVD (main responsible: DTI):

In order to navigate better in the R&D Track E, dealing with next generation of tool coatings, the Tribology Center has joined the FUNMAT-II consortium, which includes partners as Seco, CemeCon, Ionbond, Plansee, Impact coating and Linköping University. The outcome of this extended network provides new theoretical and experimental knowledge on the development of novel coatings for next generation of tool coatings. Besides, the PVD activities involve the development of many different types of coating systems supporting the overall PVD coating knowledge in many different directions hereunder especially:

- Different HiPIMS processes both reactive and non-reactive
- New CrN HiPIMS coatings
- New TiCrAlN HiPIMS coatings
- Test of alternative target compositions and possibilities
- Black electric conductive coatings for conductive and non-conductive applications
- Production scale-up and validation on large PVD units
- Homogeneity Modelling of PVD processes
- Pure metal layers.

Electroplating (main responsible: DTU):

Electroplating has focused on activities increasing the knowledge of a number of plating systems supporting both ongoing R&D Tracks and new fundamental electroplating activities such as:

- Passivation of Zn coatings
- Ag/C coating processes in a PhD project
- Cu/Ag coatings
- Barrier layer for thermoelectric materials
- Alternative to chromate passivation of aluminum
- Fe-C coatings in a PhD project.

Hereto comes that Professor S. Ray Taylor from University of Houston has been employed for 2 months.

Paint formulations (main responsible: Hempel):

Paint/organic coating has focused on:

- R&D Track C: Development of new C5 coating systems;
- R&D Track D: New chemically resistant paint systems
- Organic coatings for external Fast Tracks e.g. Varimixer.

Overall, the work has supported several publications and a patent filed on the antibacterial effect of Ag/Cu plated surfaces.

VALUE / IMPACT

The platform has contributed with knowledge and development of different PVD recipes, formulation of different electroplating chemistries and paint/polymer formulations together with both internal partners and external companies.

It is difficult to specify the exact value of the outcome, when it comes to new employments and saved expenses. However, all the developed solutions are now, in principle, available as commercial products and solutions.

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Duration of project:
January 2016 – December 2019

Collaborating companies:
Danish Technological Institute(leader), all partners

RD Platform 3: Cross cutting platform of tests and scientific publications

SUMMARY

The R&D Platform 3 is a cross cutting platform covering both specific project needs for testing, modelling and validation as well as follow up on scientific content to many of the fundamental questions raised. This way the platform has served:

1. To provide access to advanced laboratories and to perform testing
2. To participate and contribute to specific activities
3. To explore and communicate scientific investigations derived from the project ensuring visibility and credibility making results available through e.g. publishing.

OBJECTIVE / PURPOSE OF PROJECT

The R&D Tracks are based on selected and well-established R&D core competences at the involved partners. AAU have been leading the R&D Platform 3 where focus has been on testing, validation and modelling materials and scientific reporting as seen from the bullets in the above section.

Enhanced performance and extended lifetime of critical components is challenging and the need for basic understanding and investigations has been performed with focus on mainly polymeric systems where two major challenges showed: a. the complex mechanisms in the formation of a paint where models have benefited from numerous literature date on e.g. hydrogels and b. lifetime modelling and modelling of properties for solid polymers and (nano) composites.

RESULTS & DISCUSSION

More than 25 publications in the best quartile in web of science have been published reporting the research part of the work in the platform. The titles are diverse, but the work has mainly been on understanding and modelling the behavior of a polymer in a complex Multiphysics environment where the polymer (e.g. paint) is formed under the influence of for example crosslinking, diffusion, temperature, pH, salt and mechanical loads. A paint or a solid polymer is mostly always also a composite, where for example a paint contains large amounts of functional fillers like colorants, corrosion protection, anti-fouling, lubricants, fibers and fillers to increase mechanical properties. Both Hempel and Siemens Gamesa Renewable Energy have performed testing both on their own site and on the site of participating institutions and data has been used for the modelling work in this platform as well as extensive use of data from literature has formed the foundation for novel models.

Laboratory facilities at Aalborg University

List of publications from R&D Platform 3:

1. A.D. Drozdov, J. deClaville Christiansen, Swelling-induced bending of bilayer gel beams, Composite Structures 153 (2016) 961–971
2. The Effects of pH and Ionic Strength of Swelling of Cationic Gels. International Journal of Applied Mechanics Vol. 8, No. 5 (2016)
3. The effects of pH and ionic strength on equilibrium swelling of polyampholyte gels. A.D. Drozdov, J. de Claville Christiansen. International Journal of Solids and Structures. 2017
4. Bending of multilayer nanomembranes. A.D. Drozdov J. deClaville Christiansen. Composite Structures 182 (2017) 261-272
5. Structure-property relations for temperature-responsive gels Polymer. Drozdov, A. and Christiansen, J.D. Polymer, Volume 132, 6 December 2017
6. Modeling non-isothermal viscoelastic response of glassy polymers. Drozdov, A.D. and Christiansen, J.D. Acta Mechanica, 2017
7. Swelling of glucose-responsive gels functionalized with boronic acid. A.D.Drozdov, J.deC.Christiansen. Journal of the Behaviour of Biomedical Materials 65, 2017
8. Constitutive equations for the kinetics of swelling of hydrogels. A.D. Drozdov, A .A . Papadimitriou , J.H.M. Liely , C.-G. Sanporean. Mechanics of Materials 102, 2016
9. Nanocomposite gels with permanent and transient junctions under cyclic loading. Drozdov, Aleksey; Christiansen, Jesper deClaville. Macromolecules, 2018
10. Macroporous temperature-sensitive gels with fast response: comparison of preparation methods. Drozdov, Aleksey; Christiansen, Jesper deClaville. Journal of Applied Polymer Science, 135(23),46353, 2018.
11. Apparent stiffening of a graphene nanomembrane with initial curvature. Drozdov A.D. and Christiansen J.D. AIP Advances 7, 045123, 2017
12. Mechanical response and equilibrium swelling of temperature-responsive gels. Drozdov, A.D. and Christiansen, J.D. European Polymer Journal 94 (2017)
13. A simplified model for equilibrium and transient swelling of thermo-responsive gels. Drozdov, A.D. and Christiansen, J.D. Journal of the Mechanical Behavior of Biomedical Materials Volume 75, November 2017
14. Double-network gels with dynamic bonds under multi-cycle deformation. A.D Drozdov and J.D. Christiansen. Journal of the Mechanical Behavior of Biomedical Materials 88 (2018)
15. Time-dependent response of hydrogels under multiaxial deformation accompanied by swelling. A.D. Drozdov, J. deClaville Christiansen. Acta Mech 229, 5067–5092 (2018)
16. Mechanical response of double-network gels with dynamic bonds under multi-cycle deformation. A.D. Drozdov, J. deClaville Christiansen. Polymer 150 (2018)
17. Multi-cycle deformation of supramolecular elastomers: Constitutive modeling and structure-property relations. Drozdov, A.D., Christiansen, J.D. International Journal of Engineering Science 133, pp. 311-335, 2018
18. Self-recovery, Fatigue and Anti-fatigue of Supramolecular Elastomers AD Drozdov, J deClaville Christiansen Journal of Self-Assembly and Molecular Electronics (SAME), Vol. 6, Issue 1. River Publisher. 2018
19. Self-recovery and fatigue of double-network gels with permanent and reversible bonds. A.D. Drozdov and J. deClaville Christiansen. Journal of Polymer Science Part B: Polymer Physics. 2019, 57, 438–453
20. Thermal conductivity of highly filled polymer nanocomposites. A.D. Drozdov, J.D. Christiansen. DOI: 10.1016/j.compscitech.2019.107717 . Composites Science and Technology. Volume 182, 29 September 2019
21. Modeling thermal conductivity of highly filled polymer composites. Drozdov, Aleksey; deClaville Christiansen, Jesper. Polymer Engineering and Science, DOI: 10.1002/pen.25220
22. The effect of porosity on elastic moduli of polymer foams. A.D. Drozdov and J. deClaville Christiansen. DOI: doi.org/10.1002/APP.48449. Journal of Applied Polymer Science. 2019
23. Thermo-mechanical behavior of elastomers with dynamic covalent bonds.
24. Aleksey Drozdov and Jesper deClaville Christiansen. International Journal of Engineering Science IJES_2019_169. Accepted for publication, 2019
25. Micromechanical modeling of barrier properties of polymer nanocomposites. A.D. Drozdov and J. deClaville Christiansen. Submitted to Composites Science and Technology.
26. Electrical conductivity of polymer nanocomposites. Aleksey Drozdov and Jesper de Claville Christiansen. Submitted to Nature Scientific Reports on 20 November 2019

CONCLUSION

When an industrial company develops new materials or change the application of a material or the design there is an immediate need for knowledge about structure and properties of materials. Industrial applications are very complex as the service conditions and lifetime varies a lot, as does the demand for a specific property profile enabling a material to resist complex loads without failure for an often-extended lifetime. In an ideal situation, it is possible to conduct a test and conclude if a material is acceptable but with lifetime for example for a wind turbine of up to 50 years, one must rely on models to predict the expected behavior. Such models have been proposed in this R&D Platform and verified based on both measurement and data available in literature.

VALUE / IMPACT

Participating companies have benefited from novel test methods, specific data for their materials and models predicting mechanical properties and long-term behavior.

With the extensive list of international publications, the conclusion is that general knowledge has been made available not only for participants in the project but also for society.

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January 2016 – December 2019

Collaborating companies:
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Cases

R&D Tracks

R&D Tracks focus on R&D and transfer of research knowledge from the universities into development and hereby into real products. All the R&D Tracks are defined by a Fast Track industry partner with another Fast Track industry, RTO and/or university partner involved.

R&D Track A: Leading edge protection of wind turbine blades

R&D Track A1: Single Point Impact Fatigue Tester for erosion testing

R&D Track A2: Droplet path in whirling arm rain erosion tester

R&D Track A3: Detecting damage using Inline Vision System in RET

R&D Track B: Fracture mechanical evaluation of ductile Iron castings

R&D Track C: Development of new C5 coating system

R&D Track D: New chemically resistant paint systems

R&D Track E: Next generation of tool coatings for machining composite materials

R&D Track F: Antibacterial coatings development

R&D Track G: Wind loads and alternative materials in radar products

R&D Track A: Leading edge protection of wind turbine blades

SUMMARY

Leading edge erosion is one of the most critical degradation mechanisms occurring on wind turbine blades.

This project has focused on optimizing the adhesion of leading edge protection (LEP) as well as developing methods for analyzing the erosion mechanism. A method for assessing the material compatibility between two paint products for optimizing the adhesion has been developed.

A database of >60 products has been generated facilitating >600 calculated interface energy values for benchmark values. An alternative method to optimize the adhesion is to optimize the curing time of the blade topcoat before applying the LEP. The method is based on FTIR spectroscopy and more than 10% increased adhesion strength of LEP on the turbine blades could be achieved using this method. Development and advances in accelerated test methods for investigating the erosion mechanism on leading edge protection has also been performed within the frame-work of this R&D track. The three following project descriptions R&D tracks A1, A2 and A3 will explain the outcomings of this work in more detail.

OBJECTIVE / PURPOSE OF PROJECT

Leading edge erosion is one of the most critical degradation mechanisms occurring on wind turbine blades and has a huge negative impact on the annual energy production. Hence, the industry is looking for new ideas to improve the leading edge protection (LEP) and to obtain better understanding of the erosion mechanisms.

One of the important pre-requisites for obtaining a well-performing leading edge protection is to ensure a good adhesion of LEP on the blade. The purpose of this R&D track has been to develop new methods for assessing and improving the adhesion of leading edge protection. In addition, focus has been on improving the performance and insight in rain-erosion testing as well as development of a novel accelerated test method for characterizing erosion mechanisms on LEP.

Figure 1: Increased rotor blade length benefits in a higher energy production for the wind turbine, but at the same time also increases the risk of leading edge erosion. The latter being a serious challenge for the wind energy industry.

RESULTS & DISCUSSION

The work has mainly been executed by SGRE and DTU, with smaller contributions and discussions with Hempel, AAU and FORCE Technology. The main work has included:

- Development of new screening methods for assessing adhesion strength
- Development of new and/or improved erosion test techniques

The work on developing and improving the erosion test methods has been executed by PhD student Nicolai Frost-Jensen Johansen at DTU and will be explained in R&D tracks A1, A2 and A3. This R&D track presents the results obtained within screening methods for adhesion strength.

Product Compatibility Model

The adhesion of a paint on a substrate is highly linked to its wetting properties. A good wetting is often obtained when the liquid has a low surface tension and the substrate has a high surface free energy, but besides this a good match in the polarity of the two products is also needed. By measuring the surface tension of a liquid product and the surface free energy of a substrate (including their polar and dispersive parts), see Figure 2, it is possible to calculate the theoretical wettability at the interface, according to the Owens, Wendt, Rabel and Kaelble theory.

This method has been used to screen the theoretical expected compatibility between a large range of SGRE blade products and substrates as well as potential new products from external suppliers. A database of >60 products has been generated facilitating >600 calculated interface energy values. This database facilitates quick predictions of the theoretical adhesion and product compatibility of different interfaces. The method is now an integrated part of the internal product screening process in SGRE and is an important tool for screening surface cleaning methods, ageing effect, contamination errors, effect of climate conditions and not least for screening new products.

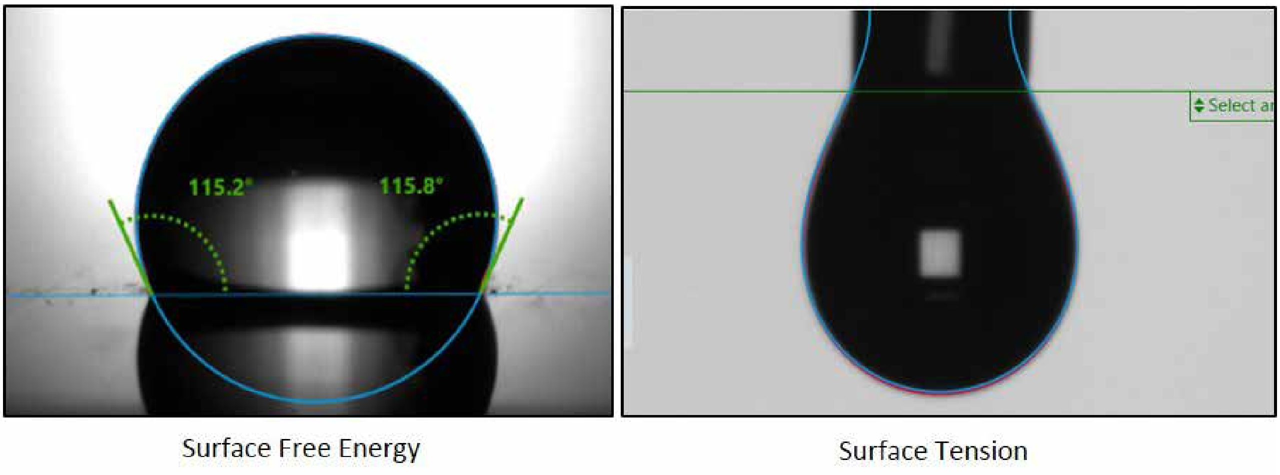


Figure 2: Left: Sessile drop method for determining the contact angle of water and diiodomethane on a substrate to calculate the surface free energy. Right: Pendant drop method for measuring the surface tension of a liquid/paint. A Drop Shape Analyzer 100 from Krüss was used for all the measurements.

An example of the use of the model can be seen in Figure 3. Here, a liquid LEP was painted on top of three different blade products (two topcoats and a primer). The adhesion between topcoat A was extremely poor with easy delamination while adhesion towards topcoat B was very good. The model predicts exactly this outcome as the interface between LEP and Topcoat B has a much higher work of adhesion and lower interfacial tension. The primer had in each case values in between.

A second example was investigated in collaboration with Hempel. In this study the adhesion of paint on a rough laminate surface prepared with peel ply (a structured polymer sheet pre-casted onto the laminate) combined with sanding and cleaning was investigated. Pull-off and surface tension measurements were used to evaluate the adhesion, see Figure 4. The results show a clear trend between samples showing adhesive failure and low surface free energy. A surface free energy of approx. 45 mN/m or above was identified as the cut off limit for achieving good bonding. This simple study showed that using surface tension data can be highly efficient and valuable as a tool to validate the surface readiness before painting – even on rough surfaces – for improving the adhesion.

Further work towards validating the model with mechanical peel tests of more interfaces is necessary and will improve the reliability of the model even further.

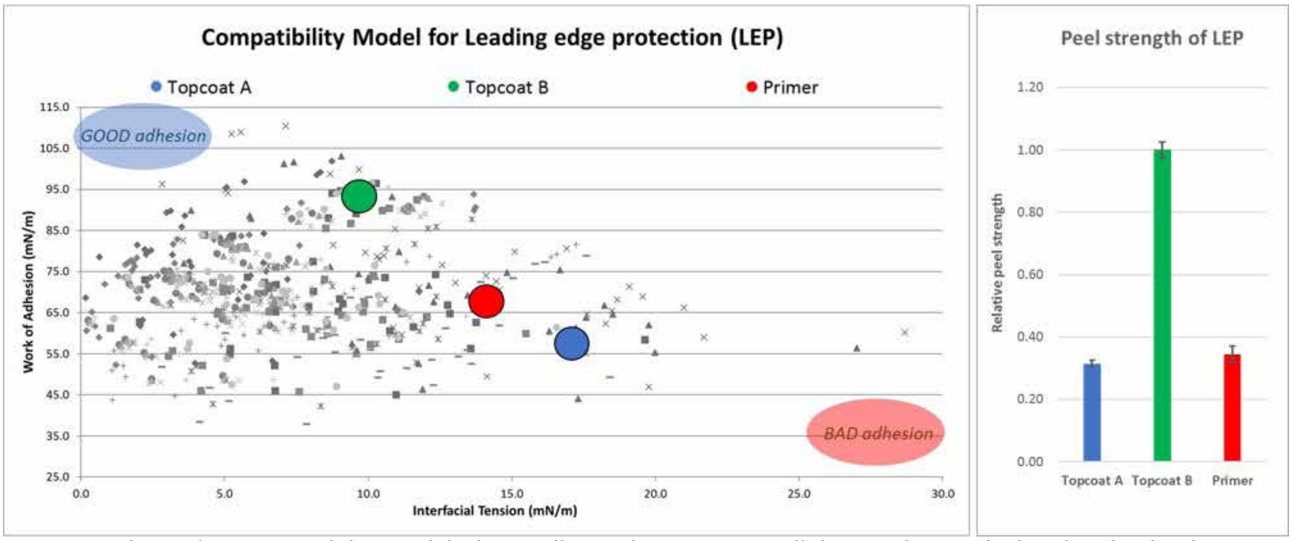


Figure 3: Left: Product compatibility model. The small grey dots represent all the interfaces calculated in the database. Good wetting/adhesion is obtained for interfaces having a high work of adhesion and low interfacial tension. Right: Peel strength of three samples of LEP on three different blade substrates.

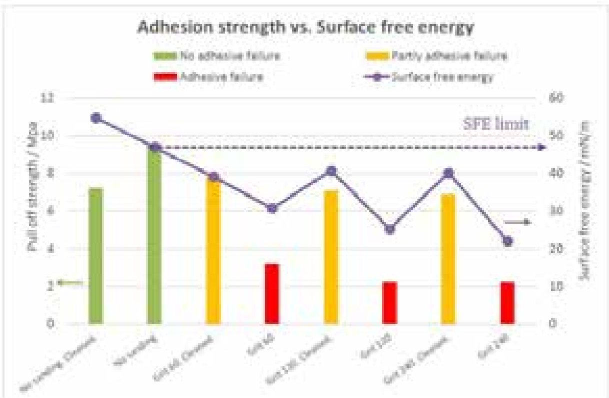


Figure 4: Pull off adhesion strength (bars) versus the surface free energy (line) for peel ply surfaces. The sanding was performed with grit 60, 150 or 240 or none. All samples were vacuum cleaned, while some of them were also wiped with isopropanol (abbreviated "cleaned"). There is a clear trend between samples showing adhesive failure and low surface free energy. A surface free energy of approx. 45 mN/m or above is necessary to obtain a good adhesion. The exact pull off value for the samples without adhesive failure is not representative for the adhesion strength as the failure occurs cohesively in the laminate.

FTIR - A tool for curing time control

The curing time of a blade coating is an important production parameter and the ability to predict, control and optimize this parameter is of great importance for production flow and product quality. Conventional methods in industry is often based on "finger print" and "sandability" tests that are highly operator-dependent test methods with significant challenges in terms of reliability and reproducibility.

An alternative, and more reliable, method to investigate the curing degree is to use Fourier-Transform Infrared Spectroscopy (FTIR). Polyurethane (PU)-based and epoxy-based coatings contain reactants that are IR active. Thus, by radiating the uncured coatings the IR-active groups within the coatings will absorb the radiation at specific frequencies. For PU-based coatings the isocyanate group (NCO) will absorb radiation around 2260 cm-1 and form a nice, single peak easy for integration, see Figure 5. As the paint cures, the peak signal will decrease and thus provide information of the curing degree. The effect of various variables on the curing time such as temperature, humidity, layer thickness, thinner content, etc. can also easily be investigated using this quick and reliable FTIR method.

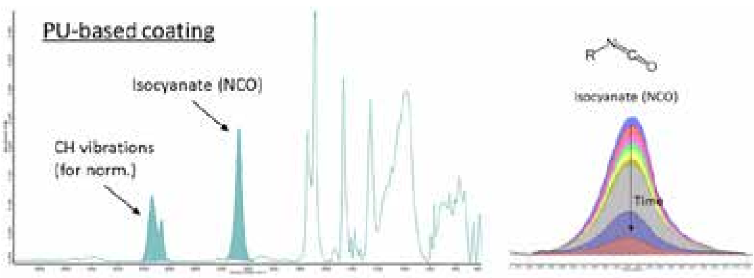
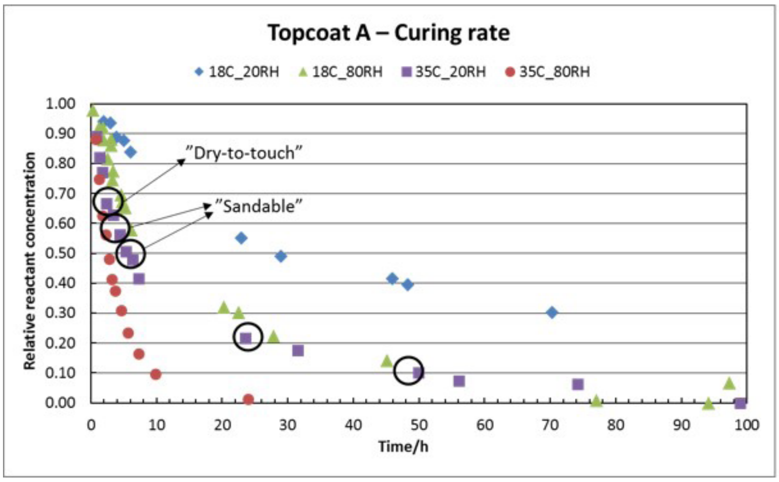


Figure 5: Left: FTIR absorption spectra of a Polyurethane-based coating. The integrated area of the reactant (NCO) peak can be used to identify how far the curing reaction has developed. The CH vibration peaks are used for normalizing the intensities of the NCO signal. Right: Illustration of the decreasing peak intensity of the NCO peak as the curing reaction develops.

Within this R&D track, we have done significant advances with this curing method. An example of how the curing rate of a topcoat is affected by climate conditions can be seen in Figure 6 (left). The resulting curing chart shows that both temperature and humidity have a significant effect on the curing time of the topcoat. The FTIR method has now become an integrated characterization tool in SGRE and used successfully on most of the paint products in the SGRE blade protection system including a new promising LEP solution.

A special feature with this FTIR method is that the information obtained from the curing study described above can also be used to investigate the "readiness" of a given painted surface for a next layer deposition. I.e., the time, at which the first layer has the optimal reactivity/curing state in order to bond more efficiently with a second layer to ensure good adhesion. To investigate this, LEP was applied onto a topcoat after various curing time intervals for the topcoat (3-48 h), according to the FTIR curing chart. In Figure 6 (right) the results show that 10% improved adhesion is achieved if the LEP is applied around 4 hours after applying the topcoat instead of waiting 6 h or more. This example shows, that also FTIR can be used to optimize the adhesion of LEP towards the blades.



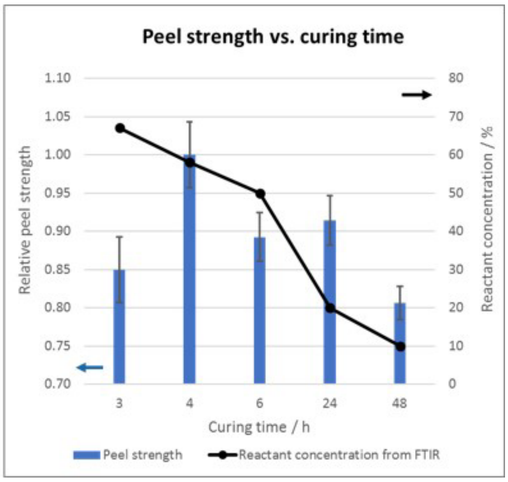


Figure 6: Left: Relative reactant concentration (integrated area of the NCO peak) vs. time for a polyurethane-based topcoat. Four different climate conditions (18°C or 35°C at 20% or 80% relative humidity). Right: Peel strength of LEP applied onto topcoat A after various curing time intervals for the topcoat (3-48 h). The peel strength is compared to the reactant concentration as determined from the FTIR curing chart.

Adhesion test techniques

To evaluate the adhesion of thin, painted layers on laminate structures is a great challenge. The traditional methods that exist, i.e. pull-off (dolly) testing, X-cut and peel-testing, all of which follow international standards, have large limitations and challenges in regard to adhesion of paints on soft laminates. The delamination often occurs in wrong interfaces and/or the fracture evaluation is based on subjective interpretation. For this reason, a large effort was put into finding a suitable method for testing the adhesion. Despite significant efforts within this project to improve the peel test methods and developing new blister type test methods, solving this task was not possible to overcome within the time frame. As both the product compatibility model and the FTIR method needs validation with real-life adhesion tests, this has been a big struggle in the project. Peel tests still seems to be the most reliable and useful method and future optimization of this could be of great benefit to many industries.

CONCLUSION

In this project we have created a product compatibility model - a method for assessing the material compatibility between two products for ensuring optimal adhesion. A database of >60 products has been generated facilitating >600 calculated interface energy values. The method is now an integrated part of the internal product screening process in SGRE and is a valuable tool for understanding how variation in interfaces, e.g. surface cleaning methods, material type, etc., can affect the adhesion and thus the overall product performance.

An alternative method for controlling the curing degree of paints has also been implemented in SGRE. This FTIR method utilizes the ability of polyurethanes and epoxide paints to absorb infrared radiation. The method is a reproducible and reliable test method, free from operator-dependent interpretations and a great improvement to conventional test methods. A special feature is that the method can be used to identify the optimal curing time of topcoats for achieving the best possible adhesion of LEP. More than 10% increased adhesion strength of LEP on the turbine blades could be achieved using this method.

Development and advances in test methods for investigating the erosion mechanism on LEP has also been performed by DTU within the frame-work of this R&D track. The R&D tracks A1, A2 and A3 will explain the outcomings of this work in more detail.

VALUE / IMPACT

Leading edge erosion is one of the most critical degradation mechanisms occurring on wind turbine blades. Each year wind turbine owners and manufacturers spend enormous billion-sized costs on repair due to failure on the leading edge. New LEP solutions and more in-depth information that can aid to the understanding of the erosion mechanism and performance of leading edge protection is therefore needed.

Although a new LEP solution has not been developed, the knowledge attained within this R&D track can aid in optimizing the production parameters and methods in order to ensure a better adhesion of the LEP to the turbine blade. An important pre-requisite for obtaining a blade with better erosion resistance and thus reduction of costly on-site repair work and down-time. The screening methods has been developed to a TRL-level of 7-8 (Technology Readiness Level) and is already implemented in SGRE product qualification program and is being used as a fundamental tool for screening new products and optimizing cleaning methods for improved LEP performance.

The development of a novel accelerated erosion test method (SPIFT) has also been successful together with improved data analysis tool for rain erosion testers. Information that is highly valuable for getting more trustworthy data for LEP performance tests. Especially important for wind energy industry and paint suppliers that rely on such erosion test methods for their R&D work within LEP development. The work on erosion test methods has led to the publication of 3 peer-reviewed articles and presentations at international conferences.

Collaboration with the other project partners has also been of great value. Sharing knowledge and cross-collaboration within technological challenging fields such as thin-film adhesion testing and erosion testing is highly beneficial for all partners to deliver quality research and avoiding redundant work.

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Duration of project:
February 2016 – March 2019

Collaborating companies:
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R&D Track A1: Single Point Impact Fatigue Tester for erosion testing

SUMMARY

This project is part of R&D track A – Leading edge protection of wind turbine blades. During the project, a new Single Point Impact Fatigue Tester (SPIFT) has been developed as a tool to simulate the rain erosion process on leading edge protection. In this method the impacting droplet is substituted by solid elastomer projectile of comparable size, speed, and density to that of rain. The advantage of this setup is that the number of impacts, speed, and position of the impacts is well controlled.

OBJECTIVE / PURPOSE OF PROJECT

Although the whirling arm type rain erosion tester is undoubtedly an excellent recreation of the conditions that can induce rain erosion in the real world, it does, however, have some limitations as a research tool. The goal of this project is to investigate/develop a complementary test that will allow researchers to probe further into the specific failure mechanisms related to this kind of material loading. Moreover, possibly to develop a tool that will allow for easy/cheap screening for new coating/protection systems.

RESULTS & DISCUSSIONS

The motivation for the development of the Single Point Impact Fatigue Tester (SPIFT), is the desire to understand the forces that a single droplet imparts to the protective coating and how this adds up over time to induce fatigue failure in the coating. The problem is that in the conventional whirling arm rain erosion tester (RET) the droplet impacts are randomly distributed over the surface (Figure 1). This makes it very difficult to compare theoretical damage models with the damages observed, as there is no way to isolate each impact.

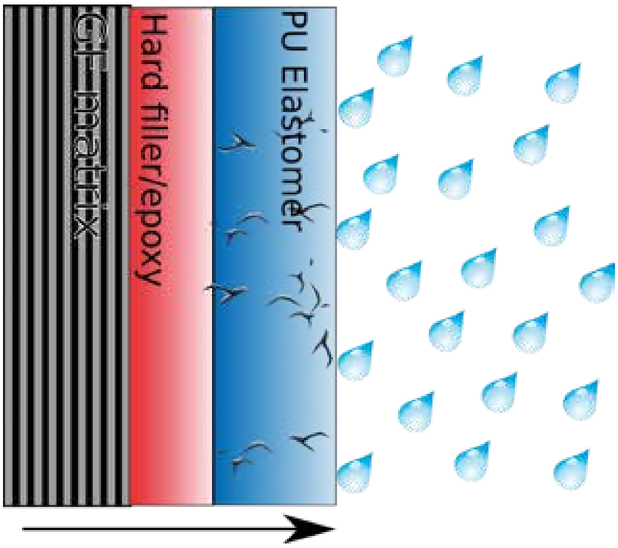


Figure 1: The surface of a coating being impacted by rain droplets

The ideal solution to this problem would of course be to impact the sample with droplets precisely at one point in a controlled fashion. However, no setup exists that is able it accelerate single droplets to the required speeds without the droplet disintegrating. Therefore the idea formed to substitute the water droplet with a solid elastomer projectile of a similar size, and density to that of the water droplet, see Figure 2.

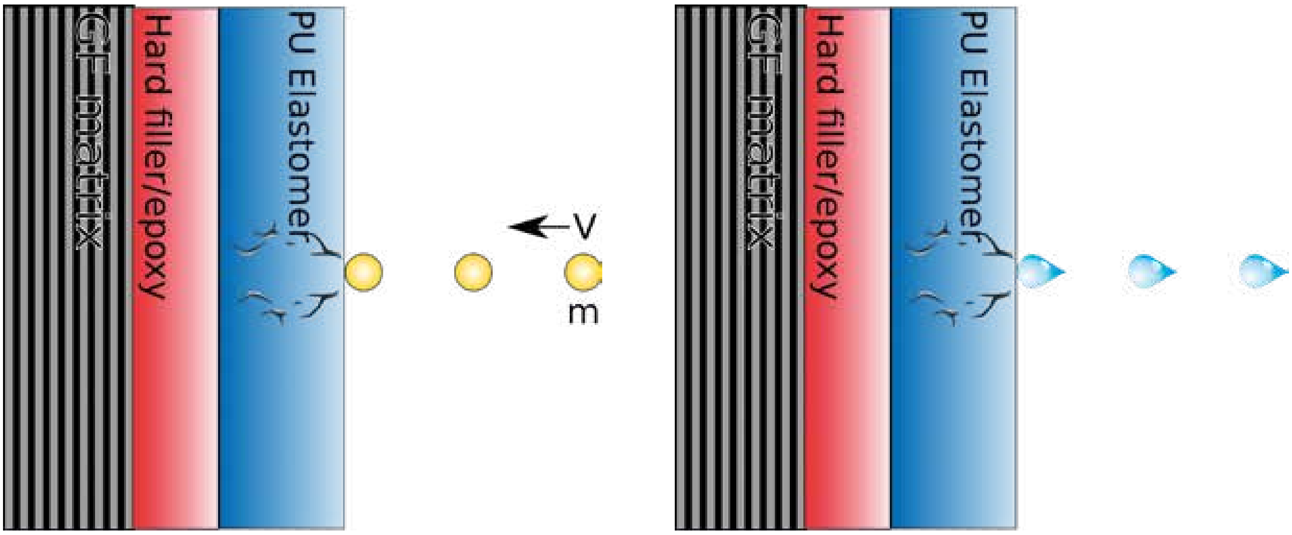


Figure 2: Instead of using impacts with rain drops, the new SPIFT tester utilizes impacts with small solid elastomer projectiles of a similar size, and density to that of the water droplet.

This idea resulted in the SPIFT tester setup as seen in Figure 3, where a computer controlled airgun firing mechanism fires projectiles at a very well-defined speed towards small flat targets.

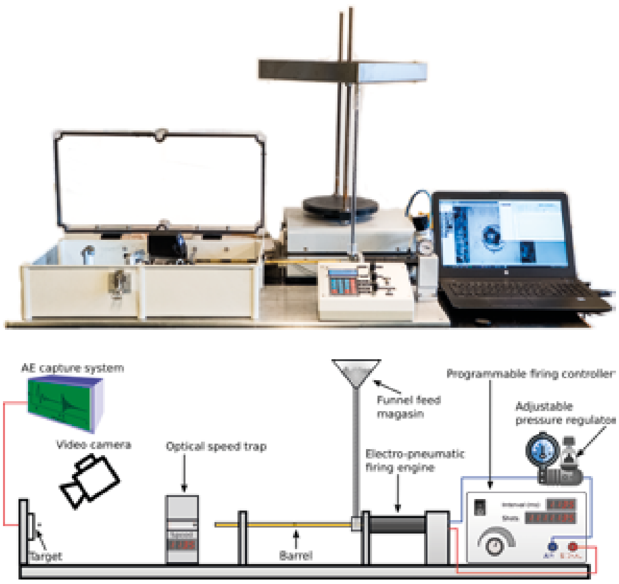
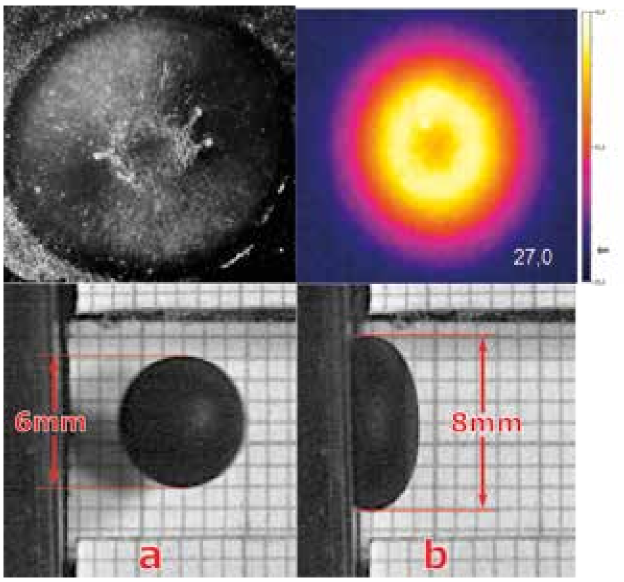


Figure 3: Single Point Impact Fatigue Tester (SPIFT) setup

A huge advantage of the setup is the ease of which different instruments can be used to probe what happens during the stages of impacts. For example, using high speed imaging to measure the duration and deformation happening during impact directly, or in situ microscopy to record the evolution of surface damage, or thermal imagining to directly measure the kinetic energy absorbed by different coatings in the form of heat (see Figure 4).

All these methods are now being used to develop the next generation of coatings, based on a much deeper understanding gained from the experiments performed on the SPIFT.

In addition, with the SPIFT setup, it has been possible to correlate the damages observed with numerical computer models. The results were published in two peer-reviewed articles [1,2].



1. Impact fatigue damage of coated glass fibre reinforced polymer laminate https://doi.org/10.1016/j.renene.2018.04.043
2. Impact damage reduction by structured surface geometry https://doi.org/10.1016/j.matlet.2018.03.146

CONCLUSION

The SPIFT setup has allowed us to perform much more in-depth analysis of the damage and failure mechanisms related to impingement erosion, and it seems possible to draw parallels between the damage observed and that observed in conventional RET tests.

We are now able to probe the effect of different material properties and their effect to a much more significant degree than before.

VALUE / IMPACT

The potential value proposition of the SPIFT can be assessed on several levels. On a simple scale, it can be used as a part of the QC-chain, to probe the performance of applied protection solution periodically to ensure consistent performance. Thus reducing the risk of a bad batch that could result in premature failure and costly recalls.

There is also the potential for use in R&D departments for use as a screening tool to cut down on expensive whirling arm testing time. Thus saving on material cost and staff hours.

Moreover, it provides value as a development tool for judging the impact of material parameters on the erosion performance and allowing for more parametric testing.

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Duration of project:
March 2017 – June 2018

Collaborating companies:
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R&D Track A2: Droplet path in whirling arm rain erosion tester

SUMMARY

As part of R&D track A – Leading edge protection of wind turbine blades, the droplet path inside the R&D A/S whirling arm tester was investigated in collaboration with DTU Wind and R&D A/S. Furthermore, the integrity of the droplets during the fall into the rotor plane was confirmed.

OBJECTIVE / PURPOSE OF PROJECT

The industry demand for better leading edge protection solutions has given rise to a need for more standardized testing procedures. This has driven the development of the new standard whirling arm tester by R&D A/S. As of 2019 about six testers have been put in commission, with the prototype being developed for SGRE. In order to have confidence in the results from the tester, it is vital to understand how droplets behave in the tester during operation. This was the goal of this investigation.

RESULTS & DISCUSSIONS

One of the unique advantages of the R&D A/S Rain Erosion Tester (RET), see Figure 1, is that it relies on the generation of a well-defined and controlled rain field. The system uses a rain generation system with non-constant rain intensity to ensure that each part of the blade impacts the same amount of water drops. The use of stainless steel needles for the droplet generation allows the droplet size to be controlled, presently to either 2.6 Ø mm or 3.4 Ø mm.

Figure 1: Rain erosion tester (RET) from R&D A/S

Droplets of these sizes are larger than what is normally found in nature, but not too different from what could occur during heavy rainfall. In most normal rainfall, the droplet size is often closer to 1.5 Ø mm. The reason for this is that the larger a droplet is, the more susceptible it becomes to aerodynamic forces.

In Figure 2, the different stages a droplet experiences as it falls into a strong crosswind are shown. It is possible to see that the droplet first flattens out into a disk, then starts to inflate before breaking apart into smaller droplets. This is a small illustrative experiment that was performed using a high-speed camera, to show the potential risks if the air in the rain erosion tester becomes too turbulent.

In collaboration with DTU Wind and R&D A/S, computer modeling of the flow inside the RET was performed to track the path and accelerations that a droplet falling inside the RET would be subjected to (Figure 3). And to further validate these models, in situ tests using high-speed imaging were performed. During the test, it was possible to track the position of individual droplets as they fell towards the spinning blade and as it was impacted.

The work was compiled and presented at the Tourqe conference in Milan in 2018, in the form of a poster, and a peer-reviewed conference paper which can be found at https://iopscience.iop.org/article/10.1088/1742-6596/1037/6/062030/meta.

Figure 2: High-speed composite imaging of a droplet falling into a strong crosswind and breaking apart.

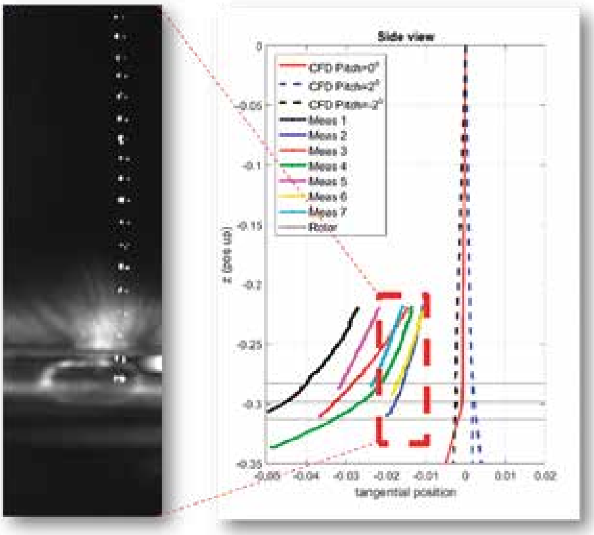

Figure 3: Computer modeling of path and accelerations of droplets falling inside the rain erosion tester, with the actual path extracted from high-speed imaging

CONCLUSION

In conclusion, it was found that droplets inside the rain erosion tester remained intact and mostly undeformed until being impacted by the spinning blade.
Furthermore, it was observed that the deflection of the falling droplets was in line with the computer models prediction for the two droplet sizes: 2.7 and 3.4 Ø mm and, this does not detrimentally affect the performance of the tester.

VALUE / IMPACT

Considering the large amount of money that both SGRE and recently Hempel spent on their newly acquired R&D A/S RET using these equipment, demonstrate that the obtained data can be relied on and is of high value. This will ensure that valuable testing time and materials are not wasted and that the testing ultimately results in better products for both Hempel and SGRE.

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Duration of project:
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Collaborating companies:
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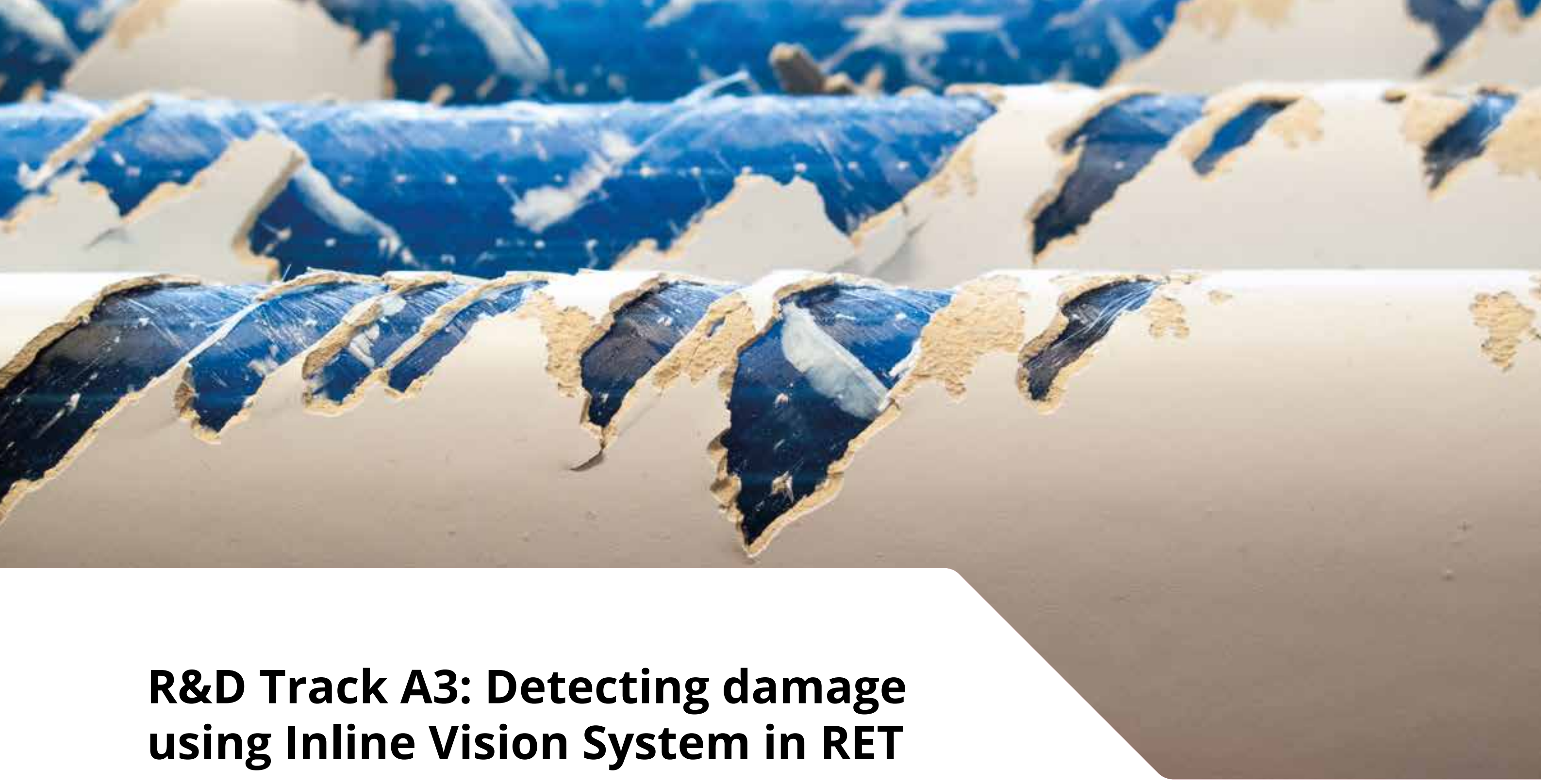

R&D Track A3: Detecting damage using Inline Vision System in RET

SUMMARY

This project was a part of R&D track A – Leading edge protection of wind turbine blades.

The focus of this project has been on how to reliably extract performance data from the R&D A/S Rain Erosion Tester, by analyzing the effect of the different parameters on damage initiation and progression. In addition to develop a new tool to help the analysis process of the data generated by this test.

OBJECTIVE / PURPOSE OF PROJECT

The industry demand for better leading edge protection solutions has given rise to a need for more standardized testing procedures. This has driven the development of the new standard whirling arm tester by R&D A/S. As of 2019 about six testers have been put in commission, with the prototype being developed for SGRE and Hempel commissioning a new tester in 2019. Getting the best possible data from the optical vision system is vital. Methods to do this in a simple way has been the focus in this project.

RESULTS & DISCUSSIONS

The primary means of evaluating the performance of the coatings being investigated is utilizing the inline vision system. This system turns off the rain field and slows the rotor down from the operational speed of 1350-800 rpm down to 5 rpm. Once this is done, each of the three blades are photographed as they pass between the two lights, as seen in Figure 1.

Figure 1: One of 3 blades in the RET tester parked between the two photo lights.

This differs somewhat from typical erosion measurements which traditionally relies on mass loss of the sample. But this approach has several limitations in regard to measuring rain erosion. Firstly, there is no convenient way to measure the weight precisely while mounted in the tester. Therefore, samples need to be unmounted before measurement, which introduces a significant amount of time to the test. Furthermore, to get the important incubation time measurement for the coating system, you would have to do a large amount of test to get a reliable number for the coating system. And last, mass loss measurements do not account for the fact that different parts of the blade have a disparate impact speed relative to the droplet, so even this information is lost.

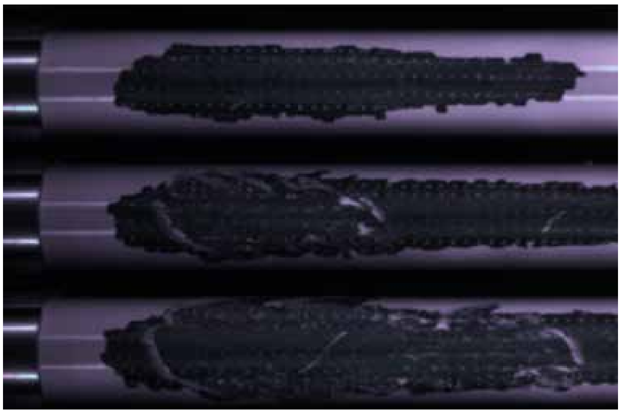

Figure 2: A set of high-resolution images taken of the blade at different time intervals in the rain erosion tester.

In Figure 2 an example of the type of data that can be obtained from the inline vision system is presented. From this set of high-resolution images, the damage progression can be followed, and it is possible to detect the very first small areas of lost coating as highlighted in the picture and illustrated below. As these areas are small and isolated damages, they express the fundamental coating performance at the local impact speed, which is a function of the position and the revolutions per minute (rpm). This means that each blade in the whirling arm tester can potentially give a multitude of incubation time measurements at a range of speeds going from high at the tip and lower towards the root, see Figure 3.

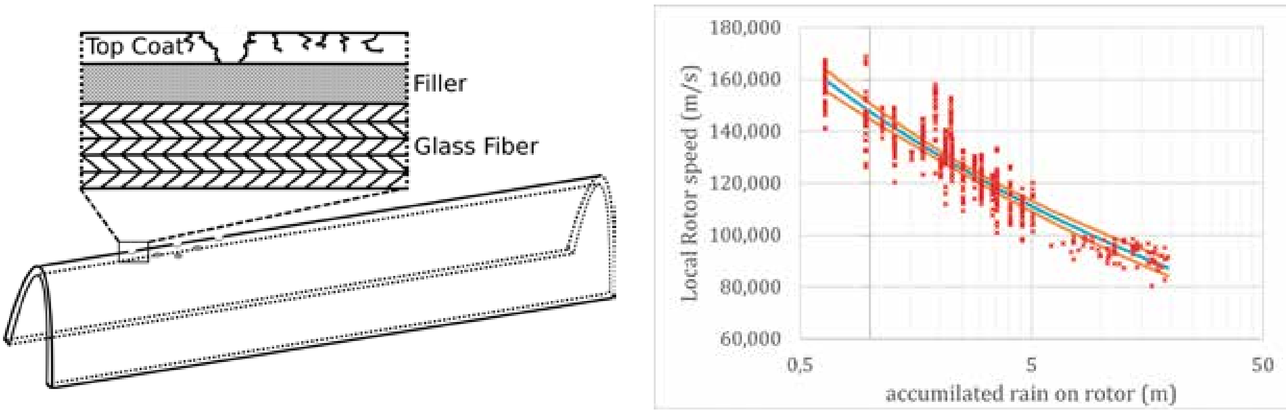

Figure 3: Left: Illustration of the leading edge of a blade with a small, isolated damage in the topcoat. Right: Lifetime data extracted from the inline vision system and plotted as a function of the impact speed and the amount of rain the coating could survive at that speed.

These investigations were performed together with R&D A/S and DTU Wind, the findings of which is described in the new recommended best practice from DNVGL-GP-0171 from February 2018. Using this method, the measured time to failure can be analysed by different parameters that equate to what an actual turbine experience, e.g. number of impacted droplets or potentially better, the cumulative column of rain impacted by a given point on the blade to failure as seen on the graph.

As the process of marking these incubation points can be daunting due to their multitude, a measuring program was developed to ease the process, which is to be used by partners to optimize the data acquisition process.

CONCLUSION

In conclusion, the project has significantly furthered our understanding of how best to use the data generated by whirling arm testers. The findings in the project has to some extent helped the industry reach a more homogenized testing procedure. And finally, also resulted in a measurement tool for use by the project partners to simplify the measuring process.

VALUE / IMPACT

Considering the large amount of money that both SGRE and now Hempel with their newly acquired R&D A/S RET spend on testing in these machines, getting the best possible data from the testing data is of very high value. And simplifying the measuring process by use of the measuring program, the load on the technicians can be reduced, allowing for the possibility of more tests at the same cost as before.

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Collaborating companies:
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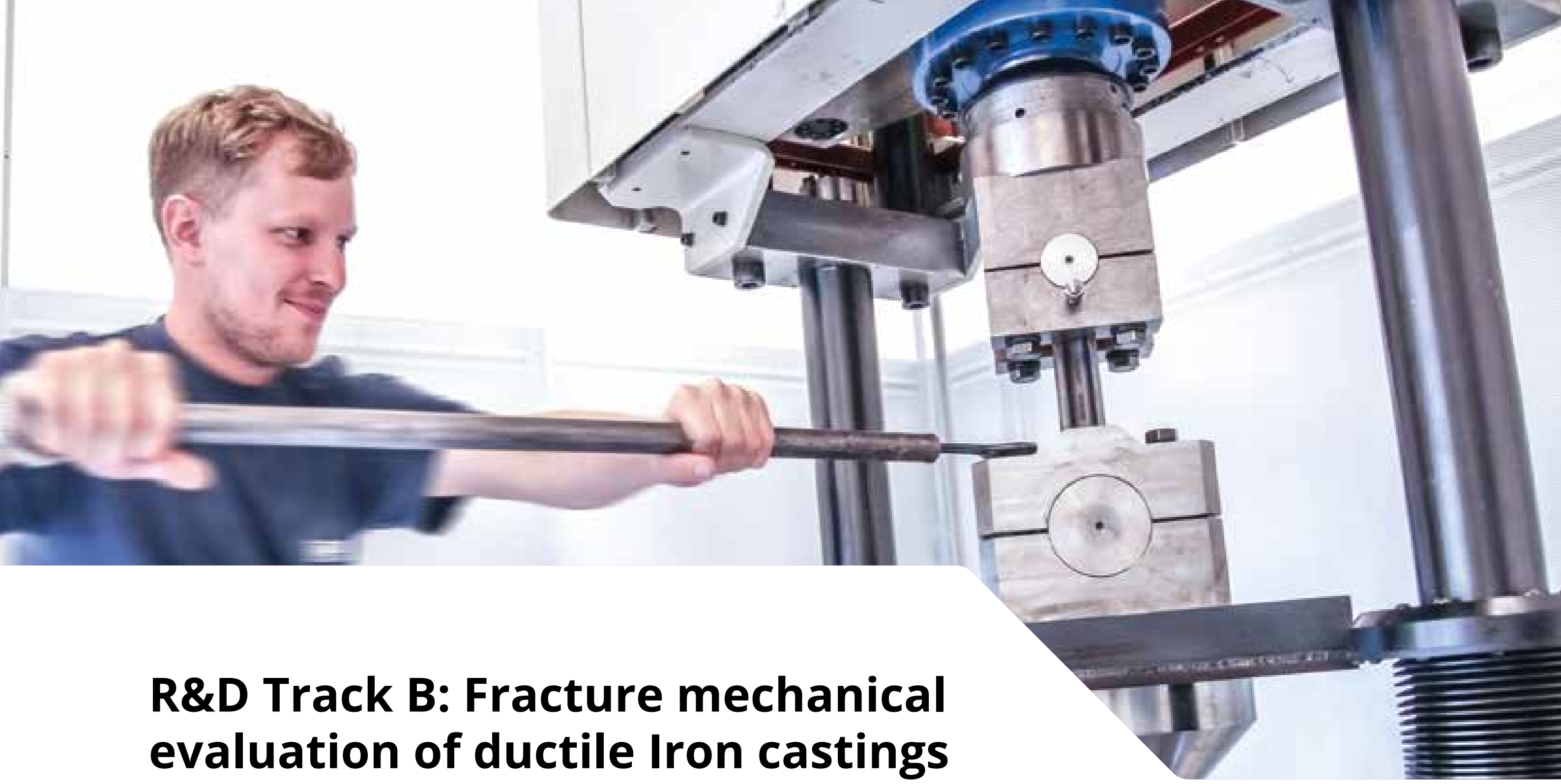

R&D Track B: Fracture mechanical evaluation of ductile Iron castings

SUMMARY

To challenge the FKM guideline "Fracture Mechanics Proof of Strength for Engineering Components" on the fracture mechanics material properties, new fracture mechanics tests are conducted. The fracture mechanics testing involves determination of the fracture toughness, fatigue crack growth curve and fatigue crack growth threshold values. Fracture toughness is determined at three different temperatures: room temperature, -20°C and -40°C. The fatigue crack growth (FCG) curves and threshold values are determined at room temperature for three different R-ratios: 0.1, 0.5 and -1. Results are compared with FKM guideline and will be used for determining new and larger critical flaw size in iron castings.

OBJECTIVE / PURPOSE OF PROJECT

- Fracture mechanics testing of ductile cast iron used by SGRE
  - Fracture toughness
  - Fatigue crack growth
  - Fatigue crack growth threshold
- Evaluate fracture mechanics test results with FKM Guideline
- Implement results in engineering critical assessment to determine new critical flaw size.

RESULTS & DISCUSSIONS

To reduce the number of disqualified large ductile iron castings, due to NDT indications of multiple defects, a fracture mechanical approach is supposed to reduce the number of disqualifications. In this fracture mechanical approach, tests will be conducted to investigate the fracture mechanical material properties of these ductile iron castings and evaluate the results against the FKM guideline. The fracture mechanical KIC test results for ductile cast irons from the FKM guideline are obtained back in 1974, and it is believed that due to better casting techniques and changed test procedures in recent years, new test results might be better than the values from FKM.

As the casted structures are of a relatively high thickness, 50 mm and above, it was decided to use Single Edge Notched Bend (SENB) specimens with width of 100 mm and thickness of 50 mm for the fracture toughness tests. The fracture toughness test was originally intended to be performed in accordance with ASTM E399 which covers linear elastic KIC fracture toughness test. The initial tests showed that the low material yield strength resulted in stable tearing and ductile fracture in these specimens, and the results were therefore conservative and invalid. Two options were theoretically possible: Increase specimen size or use ASTM E1820 which covers the KJIC fracture toughness tests which allows for stable tearing. Practically it was only option 2 which was possible, and test method was changed to ASTM E1820. With the test method changed, valid Fracture toughness results were obtained.

The Fatigue crack growth and threshold tests were carried out according to ASTM E647. Each specimen is used first to determine the threshold value, and afterwards to determine the FCG curve. For R-ratios 0.1

and 0.5, Compact Tension (CT) specimens are used. Width and thickness of the single tested specimen at an R-ratio of 0.1 was 100x50 mm. For the R-ratio of -1 Middle Tension (MT) specimens are to be used.

MTS 500 kN hydraulic test machines are used for both the fracture toughness tests, the FCG tests and threshold tests. A clip gauge is attached at the crack mouth, measuring the opening during tests. MTS software is used to evaluate the applied force and clip gauge measurements to determine fracture toughness, FCG or threshold value according to the applied standard. A test setup for a fracture toughness test with an SENB specimen is shown in Figure 1.

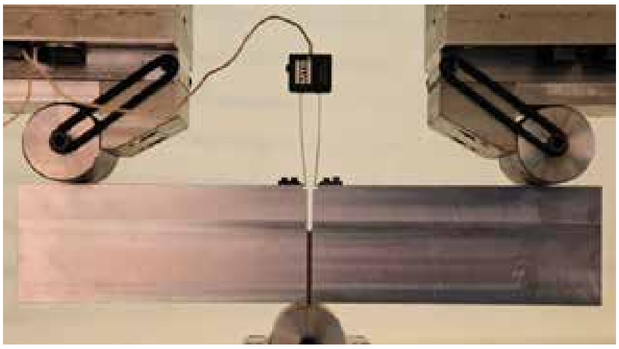

Figure 1: SENB specimen ready to be tested with clip gauge attached

Three fracture toughness tests were carried out on side grooved SENB specimens in accordance with ASTM E1820. Two tests were carried out at room temperature and one at -40°C. A J resistance curve for specimen 0138-39 is shown in Figure 2.

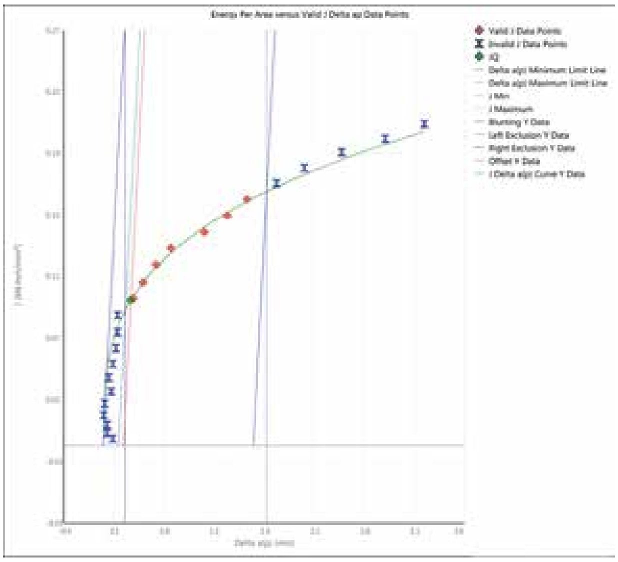

Figure 2: J-resistance curve for specimen 0138-39

The three test results for the KJIC tests are shown in Table 1

Table 1: Fracture toughness KJ results

Specimen number	W [mm]	B [mm]	B_N [mm]	Temperature [°C]	KJ [N/mm^3/2]	KJ [MPam^{½}]	KJIC
0138-39	100	50	40	20	4160	131.6	Valid
0138-40	100	50	40	20	3973	125.6	Valid
0138-42	100	50	40	-40	4519	142.9	Valid

The expected KJIC fracture toughness values were around 90 MPam^{½} at room temperature, but the obtained values of 131.6 and 125.6 MPam^{½} are close to 40% higher. This gives an indication that material fracture toughness is significantly higher than values used presently in FKM guideline. The test at -40°C gave 142.9 MPam^{½} which was not expected as the fracture toughness usually decreases at lower temperatures. More tests are required to have a conclusion on this.

One test was carried out in accordance with ASTM E647, to determine the FCG threshold value, and the FCG curve. The test was carried out on a CT specimen with a width and thickness of 100x50 mm, and an R-ratio of 0.1. The threshold value obtained was 11.0 MPam^{½}, an increase of 32% compared to the threshold value from FKM at 8.3 MPam^{½}.

The FCG curve is described by the formula $da/dN=C\cdot\Delta K^m$. The obtained FCG curve has lower a slope m equal to 5.37 compared to 6.7 from the comparable cast iron GGG-40/3 from FKM. The lower slope is not necessarily good, as it might give higher crack growth rates at lower loads.

The FCG curve with threshold value is presented in Figure 3, where the curve and threshold value of the tested cast iron is to the right of the FKM curve, showing higher threshold value and a slower crack growth rate. At lower ΔK, the two curves are closer to each other compared to higher ΔK, showing that the gains in slower crack growth rates are more pronounced at higher ΔK.

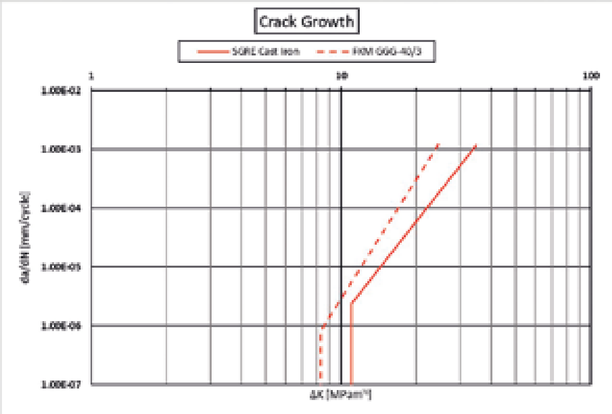

Figure 3: Fatigue crack growth curves and threshold

The constants for the FCG curve for the FKM GGG40/3 and the SGRE cast iron are shown in Table 2.

Table 2: Parameters for FCG curve and threshold values

	C MPam½	m	ΔK_{th} MPam½
FKM GGG-40/3	5.80E-13	6.7	8.3
SGRE Cast Iron	6.05E-12	5.4	11.0

VALUE / IMPACT

Research into the fracture toughness of cast irons, may in the long run reduce costs in manufacturing larger structures, as larger indications of defects may be allowed. In quantified numbers, experience shows that around 13% of large cast iron components for wind turbines are discarded due to cast defects. By implementing the new fracture mechanical properties from the initial tests, the number of discarded casting will be reduced to 5%. The end result will be a lower cost in production of wind turbines as fewer castings will be discarded.

CONCLUSION

The SGRE ductile cast iron has been tested according the ASTM E1820. The cast iron has shown significantly better fracture mechanics properties compared to the similar cast iron in the FKM guideline. The initial two tests at room temperature show an increase in KJ value around 40%. The test at -40°C show higher fracture toughness than at room temperature, which is unexpected. Further tests must be performed to investigate this issue.

The FCG threshold values and FCG curve is determined from a single CT specimen tested in accordance with ASTM E647. The initial test result shows an increase in threshold value of 32% compared to FKM guideline. The FCG curve is determined from the single specimen indicates that the material has a slower crack growth rate than what is presented in FKM.

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May 2016 – January 2019

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R&D Track C: Development of new C5 coating system

SUMMARY

Paint both protects and provides a new aesthetic impression. Paint is applied to many different surfaces (technically called substrates) like structures such as ships, bridges, buildings, vehicles, and oil & gas. The paint on the steel substrate and free-standing paint film is the main focus of the present investigation.

Current coating systems for offshore installations and bridges are in most cases based on epoxy resins in the primer and intermediate layers and polyurethane resins as UV-resistant topcoats. The established lifetime is around 30 years with regular maintenance at exposed areas such as welding seams and joints. The breakdown mechanisms are manifold, but the most important ones are connected to embrittlement due to long-term ageing under thermal cyclic conditions, combined with humidity, UV light and salt.

At some point during the life time of the paint, micro cracks, water ingress and chlorine diffusion to the interface cause corrosion of the underlying steel substrate. Hempel is frequently faced with customer demands requiring coating systems with 50-80 years lifetime expectancy.

The choice and selection of the exact coating systems are in these cases based on experience since little is known on the exact chemical and physical changes of the polymeric coating film in the final part of their lifetime. In the literature, the term "endothermic relaxation" is used to cover ageing effects but the underlying mechanism is still unknown.

OBJECTIVE / PURPOSE OF PROJECT

The purpose of this R&D track:

1. To understand the underlying degradation mechanism by using mechanical and electrochemical analytical techniques
2. To improve or develop a new C5 coating system
3. To perform accelerated test for estimating performance of coatings such as salt spray and environmental ageing according to Norsok system 1.

RESULTS & DISCUSSIONS

Electrochemical characterization of coating films

Understanding the film formation is crucial as it gives information of the quality of the curing. In-situ monitoring of coating curing was done by embedding interdigitated electrode pattern in the coating (application of paint on top of a sensor), and continuously monitoring the frequency response of a sensor. An example measurement is shown in Figure 1.

Both, the low frequency and high frequency response was used for understanding of the coating cure process as well as the benchmarking between different coating formulations.

The in-situ curing shows the rate of film formation, given in the slope. Furthermore, it is shown that the crosslinking of the 600 μm is never achieved, nor is the curing achieved for films of thickness 600 μm. All these experiments were performed at Hempel in collaboration with DTU-MEK with idea inspiration from Terma.

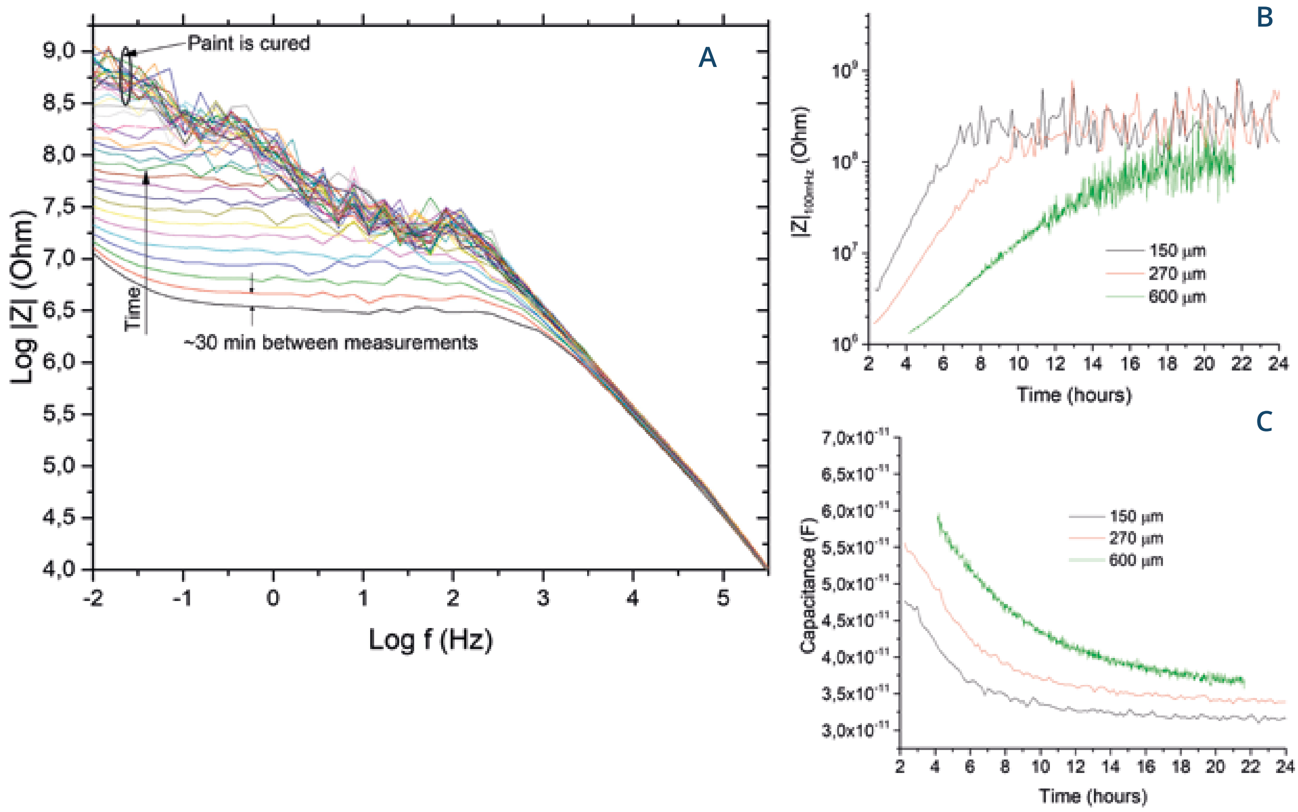

Figure 1: a. Example of EIS measurement as coating cures, and b. shows curing at low frequency and c. shows curing at high frequency (capacitance), during initial stage of curing (24 hours).

Chemical and mechanical characterization of coating films

Differential Scanning Calorimetry (DSC), Dynamic Mechanical Analysis (DMA), Thermogravimetric analysis (TGA) and Tensile Strength are the most widely used techniques that provide a wealth of information on thermal events during curing reactions (kinetic transitions), glass transition temperatures (Tg, thermodynamic transitions), change in modulus and solvent evaporation. These thermal events lead to change in the viscoelastic behavior of the coatings. As the curing process progresses the cross-link density increases along with the development of glass transition and so does the elastic behavior. Therefore, Tg and modulus are important indicators of the elastic character of the coating. The signatures or footprints acquired from single solvent born epoxy coatings with different volume solid percentage showed higher Tg, higher stiffness and high modulus for high volume solid coatings relative to the low volume solid coatings. Similarly, a system with three coatings showed the same trend. All these experiments were performed at Aalborg University's lab facilities.

On demand and in collaboration with external partner we are developing an isocyanate free water-repellent polyurethane coating. The core idea is to switch from a traditional 3 coat system to a 2 coat system. Among several prototypes, we selected four coatings to investigate the curing behavior, solvent retention and tensile stress properties at Aalborg University's lab facility. An extensive study has been carried out to measure the physical properties of the selected prototype coatings by using DSC (Figure 2), TGA and Tensile stress measurements. The graph in figure 2 shows that the coating with red curve has higher Tg and stress as compared to the rest of the coatings.

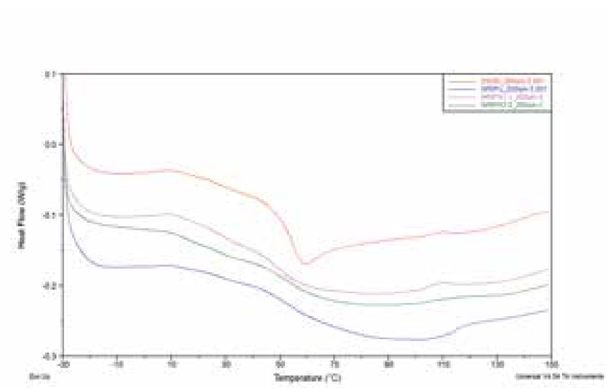

Figure 2. DSC graph of 4 different coatings.

CONCLUSION

In this R&D track, we have investigated several epoxy coatings that are presently in the market. This investigation was carried out by using mechanical and electrochemical analytical techniques such as DSC, DMA, TGA and EIS. The results obtained from these techniques showed that properties of the paint film depend on the level of internal stresses inside the coating. These techniques also provide a wealth of knowledge of how chemistry derive curing and how curing and solvent loss leads to internal stresses.

Based on the fundamental knowledge acquired from the developed epoxy coatings we are applying these techniques and knowledge to speed up the development process of new C5 water repellent coating.

VALUE / IMPACT

The focus of the development of this new improved paint system is to take a major step in developing a new system rather than taking a less incremental step. This is done by looking at the total cost of ownership for the customer by using this new paint system. This is to be used as the unique selling point. Hempel sells for about 800 million € / year in this market segment, with a market share of 7%. If this market share can increase to 10%, this will mean additional sales for 350 m € / year.

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R&D Track D: New chemically resistant paint systems

SUMMARY

The chemical industry, especially oil production is interested in cost efficient alternatives to corrosion resistant alloys. In this context carbon steel alloys can be used if surface is lined with appropriate polymer material in the form of vulcanized rubber sheets, polymer impregnated glass fiber mats or a type of coating. Several polymer materials are suitable, such as fluorinated polymers that are very resistant towards most aqueous and organic chemicals. However, application and curing of these materials require heating or irradiation and are thus difficult and expensive to install in larger vessels and reactors.

OBJECTIVE / PURPOSE OF PROJECT

The current R&D track evolves around the development of a prototype coating technology that provides very high chemical resistance at elevated temperatures and pressures. The overall success criterium is that the developed solution should be applicable as a normal coating. Thus, application by normal spray, brush and roller and should also dry and cure without external heating. Naturally the coating should resist the exposure environment that is the target for the project. A more comprehensive list of must have features are seen to the right:

- 100% reactive
- Resistance to sweet and sour crude oil and mixtures with water ISOTHERMAL) up to 150ºC
- Resistance to sweet and sour crude oil and mixtures with water (THERMAL GRADIENT) up to 120ºC
- Boiling water resistance
- No thermal stress cracking
- Tolerant of curing at high relative humidity's (bloom)
- Airless spray application
- Simple scheme application
- Minimum application temperature 10ºC
- Direct to metal -no holding primer needed
- Return to service within 72 hours
- Pass autoclave test with Alkanolamines (AMSS-067, APCS-27)
- Pass Norsok System 3F (>130ºC), Aramco APCS 27, Shell LT9 and similar customer test protocols
- Globally registered raw materials
- No HSE concerns.

The above must have criteria obviously poses some limitations that will be addressed and discussed in the next sections of this project.

The initial customer target for the coatings developed from the prototype technology sought in this project are mainly from the oil and gas sector; selected end uses are shown below:

Oil and Gas Downstream

- Customers focusing primarily on insulated downstream process equipment internal surfaces such as refinery desalters, amine absorbers columns, amine reboilers, gas dehydration columns, glycol reboilers, sulphur pits and transport equipment, some elevated temperature storage tanks operating at > 120ºC

- Customers focusing primarily on uninsulated downstream process equipment, internal surfaces such as refinery desalters, amine absorbers columns, amine reboilers, gas dehydration columns, glycol reboilers, sulphur pits and transport equipment, some elevated temperature storage tanks operating at >90ºC

- Pipework internal surfaces for process equipment operating at > 120ºC (no cold wall) or 90ºC (with cold wall).

RESULTS & DISCUSSIONS

Initially a market survey was carried out to identify commercial products with the required performance profile. A few products from companies like Belzona, Durapol and advanced polymer coatings were identified that should be able to pass at least the end-product performance part of the must have features. However, they fall short on a few other critical points among them like curing tolerance at high humidity and resistance to thermal stress cracking.

Furthermore they are difficult to apply and require a specialist crew and also have a content of unwanted compounds from a HSE perspective. Moreover, the products suffer from blooming and blushing and can only be applied as single layers. Multiple layer applications require intermediate wash and abrasive sweep blasting.

All the identified products are epoxy based with various modifications and are thus known as hybrid-epoxies. Other chemistries were briefly investigated such as vinyl-esters which have extremely good chemical resistance and track record. However, vinyl esters are not resistant to water above 90-100°C, especially at high pH. This is most probably due to hydrolysis of the ester group. Fluorinated chemistries like PVDF were also considered but discarded early due to the solid nature of the raw materials and curing temperatures above ambient.

The binder chemistry developed must have a fairly low viscosity without the addition of more than a few percent of solvent. This is a must have feature enabling near 100% solids and thus an end film thickness of 1000-2000 microns. That is needed for long term corrosion protection under these arduous conditions.

Coating based on epoxy resins cured with amines has many of the features on the must have list but falls short on a few essential ones, namely stable resistance to crude oil / water mixtures at 150°C, especially if the water phase is slightly acidic. Also, this is extremely difficult in a solvent-free epoxy product, especially if the product needs to be resistant to blooming and blushing.

Silicones/siloxanes/silicates have many of the features needed. High thermal resistance, resistance towards acids, strong bonding to steel substrates but are labile in bases depending on the crosslinking density. Siloxanes can be made highly hydrophobic on a molecular level and this mitigates the base lability somewhat.

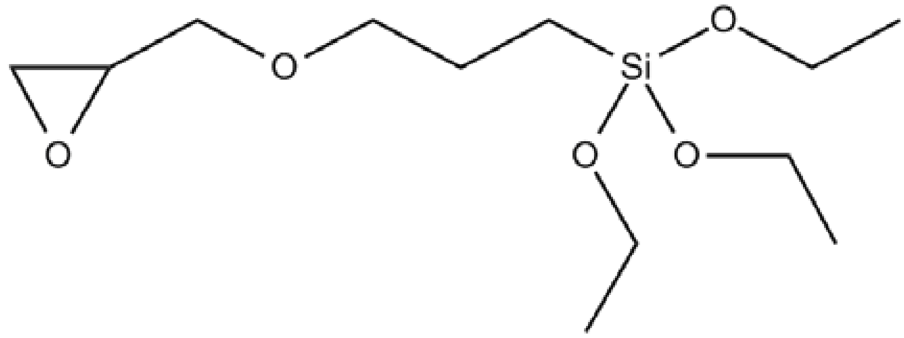
The combination of epoxy and siloxanes is well known and used as state-of-the-art corrosion and UV protection of exposed metal and concrete structures. Resins such as the Silikopon range (Ex Evonik) or similar offerings from e.g. Wacker chemicals are based on a siloxane backbone with pending epoxy groups as well as silicon-alkoxy groups. Usually various amino functional silanes are used as curing agents and the curing mechanism is based both on epoxy-amine reactions and a siloxane condensation cure.

The approximate chemical composition of the available products in the market is based on a very chemical resistant phenol Novolak epoxy resin with a ~3,8 average epoxy functionality on each molecule (such as DEN 438, ex DOW chemicals). This resin is a semisolid at room temperature and is widely used for chemical resistant coatings with a volume solids of ~60%VS, thus with a moderately high content of volatile organic components and should thus not be applicable in the current project.

Taking the viscosity into account, how are these resin types then used in solvent free coating types?

In some cases, the resin is diluted with so called “reactive diluents” which are low viscosity epoxy functional compounds such as butanediol-diglycidyl ether. However, the amount of reactive diluent that is necessary to reduce the viscosity of e.g. DEN 438 would be more than 50%. Attempting to make a final coating based on that mixture would result in a very slow curing formulation with sub-par chemical resistance as the curing rate of these aliphatic reactive diluents is low and their barrier properties are not good.

Another method to reduce the viscosity is to use monomeric condensation curing silanes as reactive diluents. Most obviously epoxy functional silanes such as the molecule shown below



Essentially the silane shown has four reactive sites, one epoxy reactive and three alkoxysilanes. The latter reacts with available water by an initial hydrolysis to the silanol. The silanol then condenses with another silanol reforming water. Thus, the net result is the consumption of water and liberation of alcohol. Another possibility is the reaction of the silanol with either an epoxy group or condensation with another hydroxyl group.

Resin formulation and curing mechanism

The use of highly functional silanes and epoxy compounds has obvious advantages in coating formulation:

- Attractive price
- Well understood regarding health and safety
- REACH registered with regards to coating formulation
- Possibility of high crosslinking density
- Large variety available commercially
- High thermal resistance
- Cures with normal amine-based curing agents used for epoxy-based coatings
- Silanes are excellent viscosity reducers.

Disadvantages include:

- Needs water to facilitate siloxane crosslinking
- Liberates methanol /ethanol during curing
- Internal stresses generated during curing
- Complete polymer structure formed during application on-site
- Difficult to overcoat due to formation of sticky layers in the surface.

What is absolutely required to form a successful coating with the required chemical resistance is the consistent formation of an interpenetrating network of silane, epoxy and amine. Thus, there can be no areas in the cured film enriched in e.g. epoxy-amine or Si-O-Si structures. As such, this is potentially difficult as the reaction rate of the different curing reactions vary differently with temperature, humidity and even film thickness. The latter due to the necessity of water availability and rate of alcohol diffusion/evaporation. The consistent interpenetrating network is important as none of the individual chemistries in the system are resistant to the range of environments the coating is potentially going to be exposed to. Thus, the chemical resistance is achieved by the fact that an aggressive chemical species might degrade a certain chemistry, but it then stopped by a resistant chemistry only a few molecular distances further in the coating. Thus, the chemistries reinforce each other as briefly outlined below.

- Amine-epoxy bonds / tertiary Amines:
  - Hydrolyzed only after oxidation
  - Oxidation is slow with certain amines
  - Stable towards bases
  - Does not hydrolyze in acids but is open to diffusion of acids due to protonation

- Epoxy-epoxy homopolymers:
  - Labile in concentrated acids
  - Stable towards oxidation
  - Stable towards bases
  - Slow reaction and liberates water during cure

- Siloxane Si-O-Si bonds
  - Hydrophobic
  - Labile to bases depending on tightness of network
  - Stable towards acids
  - Very thermally stable
  - Bonds to substrate and filler materials through X-O-Si bonds.

Having thus described the principal working mechanisms of a hybrid siloxane-epoxy binder chemistry similar to specialized commercial products the key question is:

Why is this chemistry not more widely used and how can we mitigate this by reducing or eliminating the disadvantages?

The primary issues that needs to be addressed for the project to be a success are:

- Reducing the level of mechanical stresses
- Reduce the amount of blushing/blooming/sweating/surface stickiness
- Ensure constant performance under different curing conditions.

Internal stresses in solvent free epoxy coatings mainly arise from contraction due to the chemical reaction between epoxy and amine groups. The stress is a combination of the perceived contraction of the resin combined with the increasing elastic modulus happening during curing, also physicochemical relaxation is happening to some extend during the curing reaction. The amount and type of pigments added also have a significant impact on the final stresses in the coating, mostly due to the effect the pigments have on the elastic modulus. In solvent borne coatings the internal stress is highly dependent on the film thickness and thus residual solvents left in the film after significant curing reaction has happened.

The epoxy-hybrid technology utilized in this project is somewhat in-between as there are none, or very little solvent added. However, upon curing the siloxane part of resin liberates alcohols. Theoretically, ~40 grams of MeOH are liberated from 100 grams of 3-glycidoxypropyltrimethoxysilane or ~50 grams of ethanol for the corresponding ethoxy silane. Having significant content of hydrolysable silane content in the final coating film it is straight forward to suggest that the liberation of alcohols during curing has a strong influence on the high mechanical stresses seen in these hybrid epoxy coatings. Thus, to reduce the internal stresses formed during curing the amount of liberated methanol during curing has to be reduced. The easiest way to accomplish this is to pre-react silane/epoxy mixture with an amount of water enough to hydrolyze a significant part of the alkoxysilane and let the silane / epoxy mixture pre react while distilling of the formed alcohols.

Blushing, blooming and sweating occur in normal epoxy coatings due to the reaction of free monomeric amines migrating to the surface during curing followed by reaction with CO_2 and water from the air. This reaction is most prominent at high humidity and low temperatures. Usually this is mitigated by using either polymeric amines as curing agents or using organic acids to complex with the free amines. Bisphenol A is very efficient for this purpose and is used in commercial hybrid- epoxy tank linings but has an unfavorable HSE profile. Pre-reacting the epoxy-siloxane part as suggested above could help somewhat on blooming and blushing but otherwise the amine part needs to be pre-reacted as amine-adduct, Mannich Base or alternatively an amine-siloxane. A large variety is available commercially and a rather substantive screening test should be carried out to find the right candidate.

The last issue stated above – ensuring constant performance under different curing conditions. As briefly described above, the chemistry happening in an epoxy, amine, silane system is rather complex with both hydrolysis, condensation, addition and homopolymerization reactions happening concurrently.

Further, the condensation and hydrolysis reactions are catalyzed by the amine, that in turn reacts with an epoxy group forming an alcohol. The formed alcohol can in turn either homopolymerize with epoxy or react with a silane which is also active as a catalyst for the amine-epoxy reaction. Thus, this menagerie of chemical reactions, diffusion, evaporation needs to be controlled under various reaction conditions.

From a purely chemical standpoint it would be impossible and maybe not worth the effort as it would suffice if the coating in the end has the required performance even though the chemical structures formed are different when cured under one or the other condition. As the structural analysis of the formed polymers is exceedingly difficult this is also the most practical and pragmatic way.

Thus, the way forward is to select a range of epoxy and siloxane raw materials and polymerize them under various conditions followed by curing with a few well-known curing agents. The resulting cured resin films are then inspected for haziness, sweating, crawling, formation of Bernard cells etc. Furthermore, various thermal and thermomechanical analysis techniques and spot testing with aggressive chemicals is then used as an initial performance screening.

However, it its known that the commercially available epoxy-hybrid tank lining products can have subpar performance under certain curing conditions which is something that needs to be addressed. Pre-polymerization as suggested further above would provide a constant oligomeric starting point for the final polymerization happening in the field.

Pre-polymerization will increase viscosity and thus, resins with less reactive sites are needed as part of the polymer. This might reduce performance, but the hope is that for the purpose, it will be possible to attain enough performance.

Brief experimental description

Very much aligned with commercial products a polymerization reaction was carried out on a mixture of novolak epoxy resin (3.6 reactivity, DEN 438 ex DOW chemicals) and 3-glycidoxypropyltrimethoxysilane using an organometallic catalyst (base on either tin or titanium). The reaction was carried out in a glass reactor under inert atmosphere. The mixture is heated, and a sub-stoichiometric amount of water is added along with the catalyst and heating is continued while methanol is distilled off and collected. Initial experiments along these lines did not result in a satisfactory result regarding compatibility with various curing agents as well as viscosity.

Further experimentation results in a resin based on a high functional solid epoxy resin normally used for electronics encapsulations mixed with 3-glycidoxypropyltrimethoxysilane and another multifunctional silane to increase the crosslinking density. Conventional liquid bisphenol F epoxy resin was also added to reduce end viscosity. Polymerization methodology was done as above.

Initial curing tests were done using the main group of curing agents normally employed for epoxy-based tank linings:

1. Aromatic amines – sweating and blushing in cold and humid conditions
2. Amine siloxane – Excellent properties but high cost
3. Aliphatic amine – Excellent properties with acceptable costs
4. Tertiary amine – Very brittle and fast
5. Cycloaliphatic amine – Film properties acceptable; short gel time.

From these, aromatic amines were just tried as a reference. Aromatic amines are largely not used in coating anymore due to being labelled as carcinogen.

From the above, a suitable commercial amine was chosen, and the stoichiometry was adjusted using thermal analysis methods. The aim was to find a balance between a low coefficient of thermal expansion (CTE) and a high glass transition and decomposition temperature. The latter is important due to chemical and thermal resistance and a low CTE ensures that no thermal stresses arise during thermal cycling.

Final coating formulation

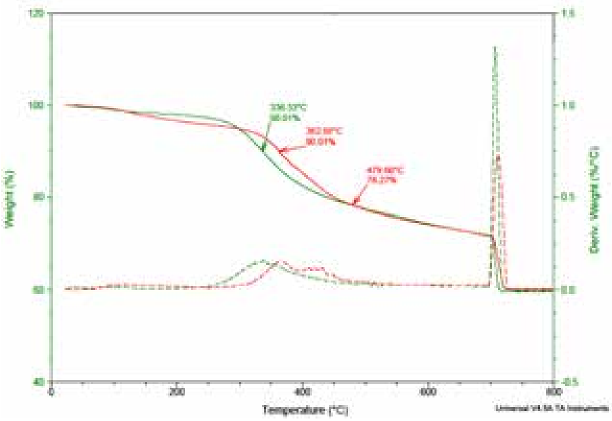
A resin is not a coating without pigments and rheological additives. For chemically resistant coatings a flake like pigments with very high chemical and in this case, thermal stability is most wanted. Thus, pigments like micaceous iron oxide, mica, quarts or wollastonite are widely used. Bayrite is also traditionally used for tank linings with success but was disregarded in the current coating as there is very little bonding between the surface of bayrite particles and the silane content of the resin due to lack of hydroxy groups on the inorganic surface.

As the resin has somewhat higher viscosity than the mostly monomeric mix in commercial formulations, choosing pigmentation with a low surface area and thus a corresponding low effect on final viscosity is important.

A mixture of micaceous iron oxide with a well-defined flake size with wollastonite was chosen. The latter pigment was used in two different surface treatments, one with epoxy silane for use in the base and one with amino silane for use in the curing agent. In principle with the amount of epoxy-silane added into the resin it should not be necessary to use surface treated pigments as good coupling would presumably happen in-situ. However, due to the reason describe above with having constant performance, using surface treated pigments removes a complete set of reactions that needs to happen during curing and thus the coating should yield more constant performance under various curing conditions.

Rheological control was accomplished by using amide waxes and fumed silica.

Initial thermal resistance was tested with thermogravimetric analysis (TGA) vs a state-of-the-art commercial reference. See below where the prototype is shown in red and the reference is green.



Below is an excerpt of the recorded thermal data on the prototype and the reference:

Sample	Exotherm onset (degC)	Exotherm peak (degC)	Tg (degC)	CTE(10-6/K) thermal expansion	10 % decomposition temp(degC)	Dry time Stage 3
Reference	57.04	110.68	84.86	68.2	336.26	4 hrs 30 mins
Prototype	56.44	109.98	65.21	140.56	362.68	2 hrs 45 mins

The CTE is somewhat higher and the Tg is somewhat lower. The CTE difference is most likely due to the amount of pigment present in the final film (PVC/cPVC ratio) which is markedly higher in the reference. Getting a higher Tg should be possible by fine tuning of the curing agent.

Finally, the cured coating was spot tested with 34% HCL, Xylene, Skydrol, MEK and IPA with no softening, discoloration, wrinkling etc.

Further, an extensive testing procedure using autoclaves and atlas cells according to NACE TM 0185 and NACE TM 0174 was planned. Uncertainties in the market situation and strategic considerations ended the project at the current stage.

CONCLUSION

A promising prototype for at HTHP tank-lining for crude oil production and refinery equipment was developed. The prototype tank-lining has attractive properties regarding application, curing and health and safety. The issue with methanol evaporation during curing has not been overcome and is at the current time not a legislative issue, it is however, not unlikely that it will become an issue in future legislation.

The coating can be re-coated without intermediate cleaning and sanding and has a reasonable curing time and potlife and thus behave much like a standard epoxy tank-lining, albeit being somewhat viscous.

VALUE / IMPACT

Coatings based on the developed technology should have better chemical and possibly also better corrosion resistance than coatings currently on the market, while still having reasonably application properties. The latter is important as the coating is then usable for a wider selection of companies and coating applicators. Coatings based on this industry should find use in e.g. biomass conversion industry as well classic industries such as oil extraction and refining.

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Duration of project:
January 2016 – September 2017

Collaborating companies:
Hempel A/S, FORCE Technology

R&D Track E: Next generation of tool coatings for machining composite materials

SUMMARY

This R&D track develops coatings for increased productivity in Danish industry. In the project focus has been on the development of a TiB_2 coating for cutting tools used in non-ferrous metals and highly abrasive composite materials. This has been performed with an industrially new technique called HiPIMS (High Power Impulse Magnetron Sputtering).

Additionally, low-friction diamond-like carbon (DLC) coatings with improved fatigue properties for machine components have been developed, where a life-time increase of up to a factor of 10 has been observed. Finally, also coating development of Ti-SiN-based coatings has been initiated. Based on these findings an international collaboration has been set up to proceed with these activities.

OBJECTIVE / PURPOSE OF PROJECT

The purpose of the project is to:

- Increase the productivity of Danish industry when cutting in non-ferrous metals and composites.
- Improve the performance of low-friction coatings for better fatigue properties to increase product lifetime in advanced material solutions.
- Explore the possibilities for making an improved tool coating for cutting in hardened materials.

RESULTS & DISCUSSIONS

Introduction

When new advanced materials are introduced into new product solutions it is of importance to enable an efficient and reproduceable production technology. Previously, a "one solution fits all" approach has been used for cutting tools as the common material selection has been limited. When more exotic materials are introduced new approaches for the production technology are needed as well. In the project, focus has been on two types of coating solutions for cutting tools.

The first solution has been TiB_2 (titanium diboride) which is known to eliminate adhesive effects when cutting in light-weight materials as aluminum and titanium. This gives a better surface quality of the final product eliminating post-processing in addition to increasing the life-time of the tool. The potential as a coating towards abrasive wear in other applications is explored as well.

The second solution mentioned, which is on a bit longer time scale, where the aim is to introduce a coating that is applicable for cutting in hardened steels, as processing in the final hardened product can lead to a significant productivity increase as several process steps can be omitted.
Common to both these coating development approaches is that a new coating technology called HiPIMS has emerged and is becoming industrially mature.

New material solutions are not only implemented in the production technology but also in new advanced components. Here performance can be improved by tailoring the surface properties independently from the bulk properties. An example of this, is when employing low-friction coatings on components leading to an increased product lifetime. Here focus is on increased hardness and temperature stability in addition to the fatigue properties as this is often a challenge for the low-friction diamond-like carbon (DLC) coatings.

Methodology

The project developments for the cutting tools have been pivoting around the HiPIMS technology. The equipment is seen in Figure 1. Previous approaches to production of tool coatings have either been based on arc technology with the drawback of many defects or based on DC sputtering where the surface quality is better. In DC sputtering, the drawback is a limited energy input in the growing film.

Figure 1: New HiPIMS coating equipment used for coating development and production.

With the advent of HiPIMS technology it has become possible to combine the advantages of both of the aforementioned technologies. By applying the power to the cathodes in very short pulses the atoms emitted from the cathode material can now be highly ionized and have a much higher energy leading to improved coating properties. This effect can be tailored by adjusting e.g. the pulse duration which is in the micro-second regime and the frequency by which the pulses occur which is typically 100-4.000 Hz. This optimization is of course within a reasonable parameter space where cooling of the target, formation of arcs etc. should be considered.

The approach to the development has been synthesis of coatings followed by analysis of their mechanical properties. As a very visual example, the morphology of selected coatings can be seen for TiB_2 in Figure 2. However, the morphology and density, of the coating are not the only important parameters. Also e.g. the hardness and elastic modulus of the coatings have been measured. In addition, it is important to assure that the coatings adhere well to the substrate so stress in the coatings has been measured as an excessive stress can lead to delamination. Finally, X-ray diffraction has been made as this can guide the development of the coatings. Based on series of depositions like these, it has been decided what the next steps should be to move towards coatings with the desired mechanical properties. When optimized coatings have been made, only application tests can tell if adjustments need to be made to have optimum performance in the applications. Feedback on the observed failures, e.g. delamination or abrasive wear can lead to a selection of different deposition parameters or further development if needed.

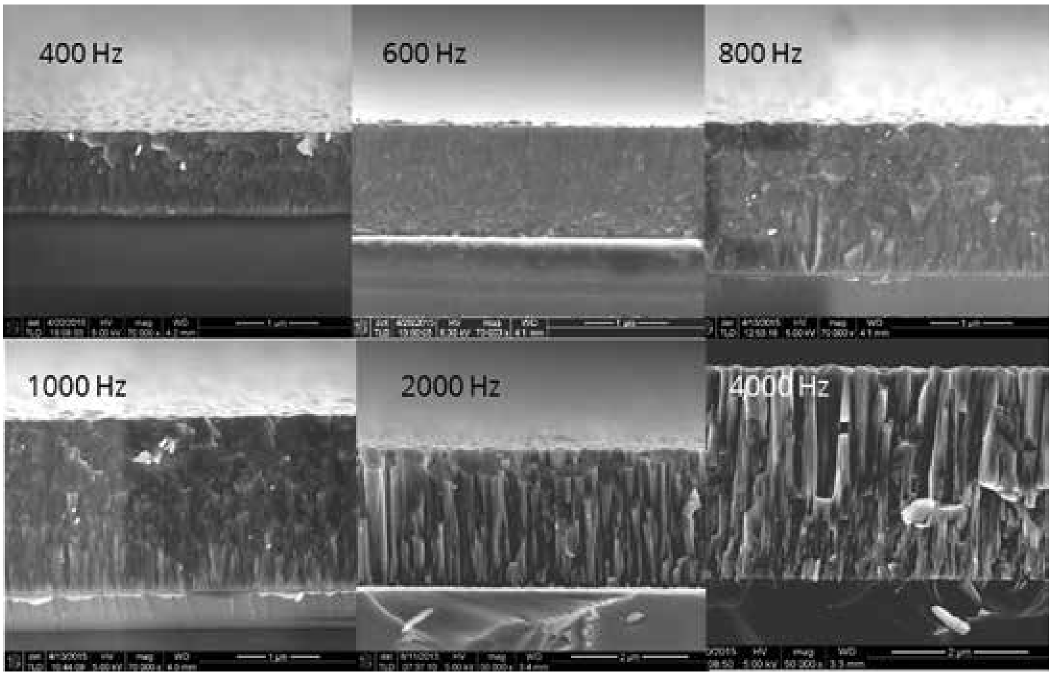

Figure 2: Scanning electron microscopy (SEM) images of cross-sections through coatings made with different pulse frequencies. Lower frequencies lead to a more ionized deposition flux. Consequently, the films at lower frequencies are denser.

In some cases, a more fundamental understanding of the problems is needed as well. This can for instance be when optimizing the low-friction DLC coatings. These coatings typically consist of an adhesion layer, a graduated layer and a top layer. Here transmission electron microscopy (TEM) is the only viable technique to have detailed analysis through the graduated layer where e.g. phase composition can be determined various places through the cross-section of the coating. Based on these findings, adjustments can be made. An example can be seen in Figure 3.

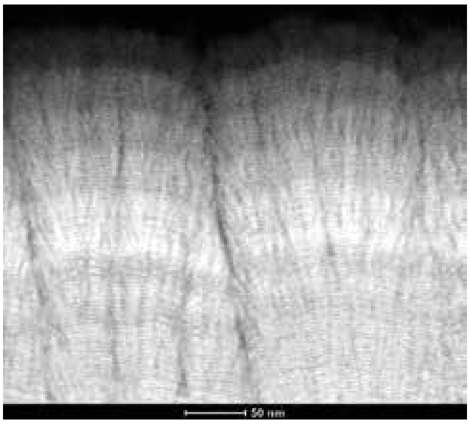

Figure 3: An example of a cross-sectional TEM image showing a fraction of the low-friction DLC coating.

As described, the path towards the commercial product goes through numerous iterative development steps. The major laboratory-based parameters that have been addressed in the development of TiB_2 are hardness and elastic modulus, stress, morphology and texture determined by X-ray diffraction.

TiB_2-results

An example of the morphology in TiB_2 was shown in Figure 2. From this, one could assume that the lowest frequencies would be best. However, when introducing data on hardness and stress as well, as seen in Figure 4, the picture is different. At the lowest frequencies the compressive stress (the minus sign on the axis shows that the stress is compressive) in the coatings increases dramatically. This can lead to poor adhesion of the coating which is detrimental to the performance. Based on the combination of all the collected data a compromise can be made where the performance is expected to be good in an application test.

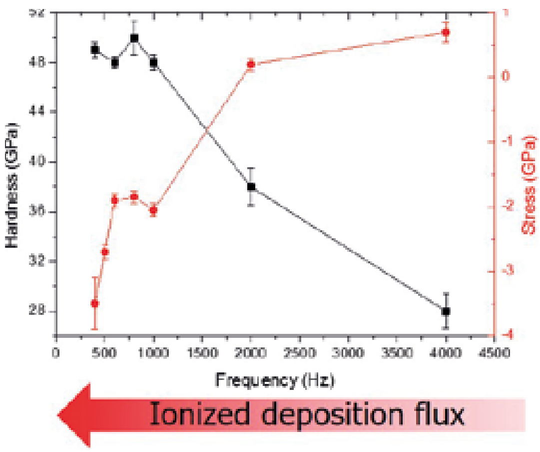

Figure 4: Hardness and stress of TiB_2 coatings as a function of frequency.

Selected candidates can then be performance tested, either in field tests or when possible in more controlled tests. An example can be seen in Figure 5, where a selected developed coating was compared to the state-of-the-art DC sputtered coating. The cutting test is made in titanium grade 5. At the current cutting length the DC sputtered coating starts to degrade at the cutting edge whereas the developed coating is still performing well without any degradation.

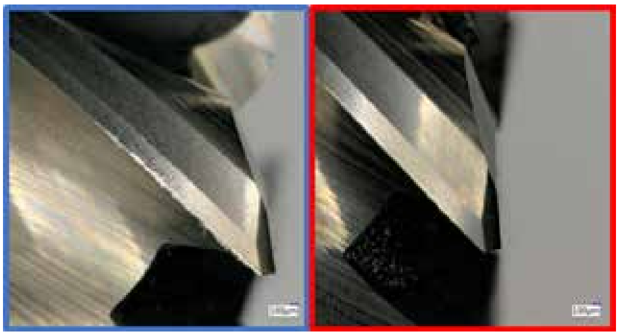

Figure 5: Example of cutting edges in a performance test where the developed TiB_2 (red) has been benchmarked against the previous state-of-the-art (blue) when cutting titanium grade 5.

At later stages in the cutting test the failure mechanism for the developed coating was explored as well, leading to smaller adjustments based on the laboratory data. This improved the performance further and is now the commercially available product.

TiSiN-results

For TiSiN coating development the status is currently that laboratory data has been made in a similar way as shown for TiB_2, but it has not yet been performance tested. An international collaboration has been set up where the performance testing and further development can be made. In addition, also new approaches to the coating production will be pursued to optimize performance. The aim is to have a product that is suitable for cutting in hardened steel.

DLC- results

The final part of the development activities has involved producing harder and more temperature stable low-friction coatings and optimizing the interface between the support layer and the top layer as failure due to fatigue issues often originate from that part of the coating.

A new Si-doped DLC coating has been developed which is markedly harder and more temperature stable than previous DLC coatings. During the project the hardness has been increased from 1200HV to above 2000HV with good adhesion. In addition to this, the temperature stability has been increased from about 230°C to 300°C in annealing tests for 2 hours. This has been determined by both mechanical testing and Raman spectroscopy. Hardness in DLC coatings mainly originate from the C-C sp^3 network, which means that a hardness test is a good indicator for the preservation of the sp^3 network. In Raman spectroscopy, it is possible to observe changes in the carbon sp^2 content, where thermal degradation would lead to graphitization.

For the fatigue properties of the DLC coatings, a systematic approach with lab scale tests is a bit more complicated. The main input for adjustments has here been inspection of failed samples from field tests and the knowledge that can be extracted from laboratory analysis like SEM or TEM, which is shown in Figure 3. The tests have been made by a company where cyclic movement in the range of millions of cycles are desired which is limiting the amount of test possibilities. Despite that, it has been possible during the project to go from failure at below 10 mio. cycles to passing 100 mio. cycles in test which was the desired goal. The findings from these tests are now being introduced into new updates of DLC coatings. The two activities with optimizing hardness and fatigue properties have led to launch of a new Si-DLC coating that is now commercially available and is being tested at several other users of DLC coatings.

CONCLUSION

- A TiB_2 coating has been developed in the project which is outperforming state-of-the-art at the project start. This has been exemplified in field tests where an improvement of 20% has been shown in milling casted Al. A 30% improvement in milling Ti grade 5 has been obtained, and additionally, better surface quality when machining Al has been obtained due to minimized adhesion between the workpiece and the tool. Also in completely other segments the coating has found use where for e.g. milling in PP with chalk fillers, an improvement of 400% in lifetime has been obtained.

- A DLC coating with increased hardness and temperature stability has been obtained, combined with improved fatigue properties. The hardness is changed from 1200 HV to >2000HV, the temperature stability has increased from 230°C to 300°C and in cyclic tests, an improvement from 10 mio. cycles to >100 mio. cycles has been obtained. This product is now commercially available.
- Development of a TiSiN coating for cutting in hardened steel has been initiated. This work will be continued in an international collaboration to obtain a product that can improve the entire European production industry.

VALUE / IMPACT

TiB_2 is now a commercially available product that is produced on a regular basis. This gives of course a value to the Danish tool producers who obtain a competitive edge. In addition to this, also DTI has an income due to commercial job coating. However, the main value is generated by the users of the tools. In the cost of producing parts, the tools typically account for 3%, whereas about 60% of the cost is directly related to labour and equipment costs. This means that an obtained productivity increase will have a significant impact on the revenue in the production sector, even if the tools becomes marginally more expensive.

For the developed Si-DLC coating which is commercially available as well, the main value is generated by the users of the coatings. By using the coating of better performance, service intervals can be extended for the produced equipment. This is especially important for equipment that is exported, as short service intervals will require an even more fine-grained maintenance and service organization.

The TiSiN is not generating revenue yet, however this product has a potential to enable production directly in hardened material which can eliminate several process steps in the production. The aim is to commercialize this in a European collaboration.

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Duration of project:
February 2016 – July 2019

Collaborating companies:
Siemens Gamesa Renewable Energy A/S, Terma A/S, Danish Technological Institute

R&D Track F: Antibacterial coatings development

SUMMARY

Copper and copper alloys are well-known antibacterial agents and have recently gained more interest as materials for frequently touched surfaces like doorknobs, bathroom fixtures, tables, etc. Silver is also toxic to bacteria and has been widely used in various biocidal applications, from wound dressings to water distribution systems.

A copper-silver alloy or a combination of silver with another material, such as carbon, can therefore improve and enhance the antimicrobial efficiency of the single metals by creating an electrochemically active material. A copper-silver alloy coating can reduce and potentially eliminate the microbial contamination on furniture items, instruments, equipment in healthcare settings and food industry, whereas a silver-carbon coating on filters can reduce and prevent bacterial contamination in air and water systems in food industry and animal farming.

The consequent reduction of infections and microbial contamination will have consistent benefits from both a human and an economic perspective.

OBJECTIVE / PURPOSE OF PROJECT

An ongoing industrial PhD project financed by Elplatek A/S and Innovation Fund Denmark in collaboration with DTU has proven that it is possible to electroplate a copper-silver alloy on stainless steel and the coating has showed high bactericidal properties.

The purpose of this R&D Track is to focus on the practical manufacturing and on the feasibility of an industrial-scale production of the copper-silver alloy and the silver-carbon coatings. For that, we tried to improve the existing electroplating process and find other alternatives to the electrolyte chemistry.

Besides, the project also evaluated the implementation of the copper-silver alloy coating in potential applications, e.g. in the healthcare and food industry and found potential customers and/or investors. The silver-carbon coating has been tested in an air-cleaning configuration within the Fast Track consortium.

RESULTS & DISCUSSIONS

Copper - Silver

In a preliminary assessment of the industrial applicability of the copper-silver alloy electroplating process, the electrolytic bath was scaled up from a two-litre lab-scale set-up to an eight-litre bath. A double anode system set-up has been developed, consisting of silver and stainless-steel anodes connected to two different rectifiers. Different plating parameters were tested with respect to composition, thickness and brightness of the final coating. Moreover, the effect of the addition of different brighteners alone as well as in various combinations were evaluated by visual inspection and X-ray fluorescence spectroscopy. All the tested plating configurations and plating parameters resulted in a marginal increase of the coating thickness and a slightly brighter coating after the addition of the combination of brighteners in the double anode system.

Regarding an alternative chemistry to the cyanide-based electrolyte, we developed a pyrophosphate-based electrolytic process that allowed obtaining a coating with the desired composition, but no significant increase in the coating thickness could be observed from the measurements on the samples' cross sections. Cyanide-based plating processes allow better controllability during the electroplating of precious metals. However, a cyanide-free electroplating bath may be advantageous for reasons of sustainability and broader commerciality worldwide. For this purpose, we are currently testing pulse reverse platings with different combinations of pulse forms, trying to ensure the controllability of the pyrophosphate-based bath and avoiding precipitation of complexes.

The properties of the copper-silver alloy coating made it very attractive for various applications in healthcare settings and the food industry, therefore the manufacturing process needs to be carried out in a rational way in order to achieve and introduce a competitive coating solution to the market. The existing copper-silver alloy electrolyte can be implemented in an automated electroplating process, and this could be done on an industrial scale, however, we are still working on the other possibilities as well.

In regard to the intended applications of the coper-silver coating, durability tests need to be performed on the present coating in order to evaluate its lifetime and whether a deposit thickness greater than two microns will be necessary. The best way to evaluate both the durability and the antimicrobial performances of the copper-silver alloy coating and at the same time searching for potential customers and investors are field tests.

Therefore, a two-month field test was carried out in a private medical clinic in Kvistgaard (Denmark) in order to evaluate the antibacterial and physicochemical properties (durability, aesthetic appearance) of the copper-silver alloy coating in a real application. Results showed that the copper-silver alloy coating decreased the bacterial survival on coated door handles significantly. At the moment, another field test is running in a wound care center in Lubbock (Texas), where there is a huge interest in implementing the copper-silver alloy coating as an additional and effective control measure against spread of germs.

In the view of a regenerative design approach, we have also successfully demonstrated that the coating can be completely stripped off by using persulfate-based chemistry, and copper and silver oxide particles can be recovered and reclaimed by filtering or by electrolytic methods. In this way, the same item may be stripped and recoated once the coating is worn off. This will guarantee functionality over time and at the same time allow for business model involving a service contract.

Field test in Lubbock, Texas.

Coated and uncoated door handles during the field test in Kvistgaard, Denmark.

We are also in collaboration with ACT.Global, where they have started the testing of a jointly developed test method for evaluating the potential of the copper-silver alloy coating to reduce and eliminate the microbial contamination on equipment in the food industry that is difficult (if not impossible) to clean efficiently.

We are currently filing a patent application at the Danish Patent and Trademark Office.

Carbon-silver

The carbon-silver surface has been produced at DTU and tested by TI. The silver is deposited on carbon by reducing silver nitrate creating silver nanoparticles on top of carbon fibers having a large surface area that can work as an antibacterial filter surface.

The effect of the filter produced by DTU has been tested at TI, and the antibacterial property is well proven. Moreover, the filter structure and silver coating have been documented and show that the technique is practically useable. Initial test in an air-flow configuration is promising. The test will be redone in an environment characterized by a higher bacterial load.

CONCLUSION

The cyanide-based electroplating process allowed obtaining a highly effective antibacterial copper-silver alloy coating and it was characterized by a better controllability, but its implementation in an industrial scale would require an automated electroplating step process.

In order to increase the thickness of the deposit and avoid a multistep plating process, we are currently testing pulse reverse plating also in combination with cyanide-free bath, alternatives that may be advantageous for reasons of sustainability and broader commerciality worldwide.

Field tests are the best way to evaluate both the durability and the antimicrobial performances of the copper-silver alloy coating and tailor its thickness to the intended application. Our first field test carried out in a private clinic in Kvistgaard gave very promising results and another field test had been carried out in a wound care center in Lubbock in Texas. Results showed a statistically significant difference in bacterial survival between coated and uncoated door handles. We are collaborating with ACT.Global for testing the copper-silver alloy coating as a solution for reducing and eliminating the microbial contamination on equipment in the food industry.

The silver-carbon coating has shown promising results in the first air-flow tests.

VALUE / IMPACT

This project could potentially introduce Elplatek A/S to customers and investors in the healthcare sector and food industry. Global leading companies in the healthcare industry that provide medical equipment, supplies and services, such as NovoNordisk, Siemens Healthineers, Coloplast, Berendsen and Medtronic may have an interest in licensing the present invention.

Moreover, it will open up for a number of possibilities of collaboration with other companies in the field, such as the ongoing collaboration with ACT.Global A/S that provides long-term and eco-friendly disinfection solutions in healthcare, hotels and food production.

The copper-silver alloy coating can reduce and potentially eliminate the microbial contamination on furniture items, instruments, equipment in healthcare settings and the food industry. Silver-carbon coating on filters can reduce and prevent bacterial contamination in air and water systems in the food industry and animal farming, and also as a potential solution to treat drinking water in disaster areas.

Therefore, the reduction of healthcare-associated infections and food contamination would have consistent human and economic values.

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Duration of project:
April 2017 to April 2019

Collaborating companies:
Elplatek A/S, Technical University of Denmark (DTU) and Danish Technological Institute (TI)

R&D Track G: Wind loads and alternative materials in radar products

SUMMARY

The use of alternative materials and/or manufacturing processes when designing the future radar components, can be a significant factor to improving performance and manufacturing costs. To optimize use of materials and reduce risk of employing new philosophies in a design process, it is imperative that the loads set is valid for the specific application, as this will allow less conservatism.

R&D track G is essentially a continuation of Fast Track 3 and has focused on two parallel activities:

- Validation of fatigue wind loads
- Searching for alternative materials

Validation of wind loads

This consists mainly of a full-scale test of a rotating radar antenna which is intended to collect fatigue loads data over several months. The test data will be used for validation of computational models for predicting life-time fatigue loads. At present, the full-scale test is on-going, and results have not yet been produced.

Searching for alternative materials

This activity focuses on identifying new materials and/or manufacturing processes that can be used advantageously in radar components. It includes two innovation workshops held with collaboration partners and hosted by Terma. Using ideation methods, Terma employees engaged with external experts with great success. The outcome of the workshops has resulted in several new R&D projects.

R&D track G overview:

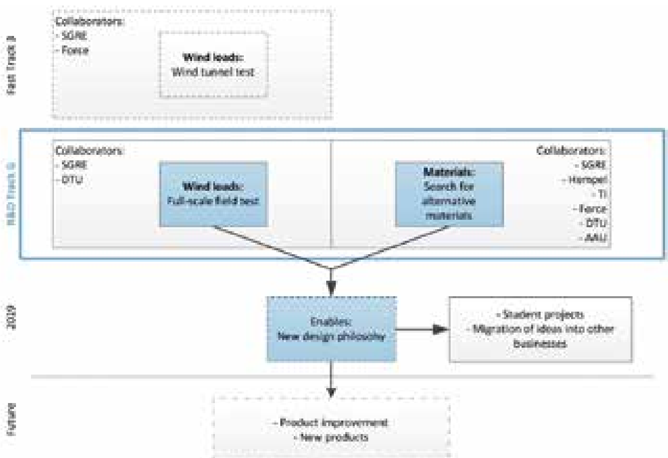

Figure 1: Overview of R&D track G

OBJECTIVE / PURPOSE OF PROJECT

The purpose of this R&D track is twofold: Validate fatigue wind loads estimation methodologies and search for alternative materials for design and construction of radar components.

- Search for alternative materials in radar products
  - Identify alternative materials and associated manufacturing processes
    - E.g. change from aluminum to composites, or change from machining to AM
  - Increase design freedom, product performance and reduce manufacturing cost

- Validate wind loads estimation methodology
  - Improve prediction of world-wide wind-based fatigue loads on radar antennas.

RESULTS & DISCUSSION

Searching for alternative materials

To generate inspiration for applying new materials and/or manufacturing processes into Termas product lines, two ideation workshops were held as an attempt to provide the best possible synergy between Terma and our collaborators in this R&D track. The fundamental idea with the workshops was, that Terma could participate with internal experts with the best possible knowledge of specific applications and products, whereas external experts, in virtue of their limited insight into Termas products, would be least restricted in their mindset.

Format of the ideation workshops:

- **Facilitation:**

  - Workshops require an unbiased facilitator. The important role of the facilitator is to control the format of the workshop and to avoid affecting the results and output of the discussions.

- **Participants:**

  - An equal number of internal and external experts from Terma and R&D track collaborators is required. This balance is essential for the host (Terma) to absorb the ideas generated by the external participants

  - An even number of male and female participants is desirable. Experience shows that this generally results in the most diverse and bountiful ideation output.

- **Idea Pitching:**

  - At the beginning of the workshop, the intended direction of the ideation is pitched with a short presentation.

- **Sparring and rotation:**

  - Internal and external experts are paired 1-to-1 and discuss ideas, problems, solutions. During the workshop, participants rotate so that all experts have a change to ideate 1-to-1.

- **Summary:**

  - Results are discussed in plenum for others to provide feedback.

Workshop 1 “Materials”:

In workshop 1 “Materials”, experts in materials, fabrication and micro-wave technology gathered and together identified ways of using alternative materials in the design and manufacture of radar antennas and waveguide components.

Topics discussed:

- Fundamentals of changing a design philosophy by changing materials and manufacturing processes. If one wants to achieve radical results in terms of production cost/time, and/or performances such as durability, weight, etc, this requires radical changes in the philosophy of designers. I.e. one cannot expect great results from taking small steps.

- Use of composites in antenna designs. Risks and opportunities of using foams in sandwich constructions in severe environments.

- Use of AM in manufacture of wave guide components, such as filters, where previously unobtainable shapes and design can now be realized.

Results:
Workshop 1 has resulted in another Fast Track project that involves manufacture and test of metal plated

AM-based polymers for wave guide components. Also, the general discussions that took place in workshop 1 has provided inspiration for continued internal R&D projects.

Workshop 2 "Fatigue and Damage Tolerance":

In workshop 2 "Fatigue and damage tolerance", experts in mechanics of solids and composites, design and manufacture of composites, additive manufacturing (AM) and materials testing gathered and discussed opportunities and challenges with respect to highly weight optimal designs in e.g. composites or 3D printed metals.

Topics discussed:

- Challenges involved in test and inspection for cracks in 3D printed metals structures, especially design based on topology optimization which can include complex organic shapes that prevent use of existing methods for crack inspection.
- How to reduce cost in the determination of fatigue and damage tolerance of composites, which traditionally involves a substantial and expensive test campaign, by applying analyses to a higher extent?

Workshop 2 resulted in an investigation of the damage tolerance (in this case "compression after impact") of a 3D printed thermoplastic with carbon micro particles. Characteristics of this printed material is to be compared to those of conventional composite laminates. Currently this investigation is on-going, and no results have been produced yet.

Validation of fatigue wind loads

Below diagram outlines a process for assessing the lifetime induced wind loads of an antenna, which is the required input for structural fatigue analysis and lifetime estimation.

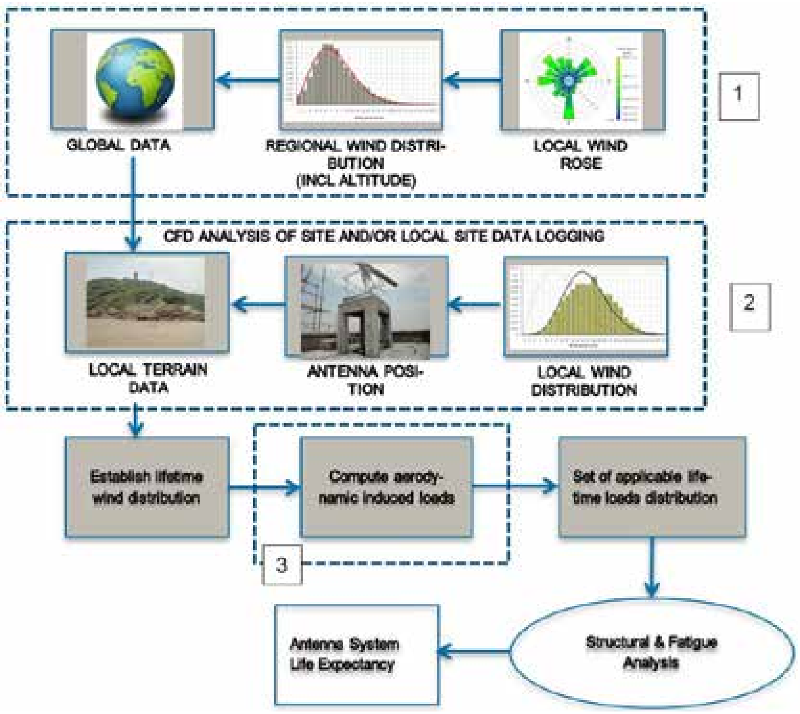

Figure 2: Assessment of the lifetime induced wind loads of an antenna

The steps (1-3) outlined in the diagram above were topics of Fast Track 3, with the following overall outcome:

Step (1): The tool WAsP was identified as a potential solution to identifying an overall wind spectrum in an arbitrary world-wide location.

Step (2): CFD analyses were used to identify pitfalls of using wind anemometers to determine wind conditions for specific radar antenna installations.

Step (3): Wind tunnel test data was used to validate the models used for wind loads estimation in uniform wind fields.

In this part of the R&D track, a full-scale test is setup to monitor wind conditions and fatigue loads for validation of the process outlined above, until the arrival at the life-time loads distribution in step 4.

Test setup:

The test setup consists of the same antenna, instrumentation and data acquisition used in the wind tunnel test for Fast Track 3. The antenna is placed in a realistic position on top of a building. In the near field of the antenna, a 3D wind anemometer is placed. In addition, a 2D wind anemometer is placed 800m away in a terrain free of neighboring buildings. The reaction loads on the antenna is measured simultaneously with the wind at both locations.

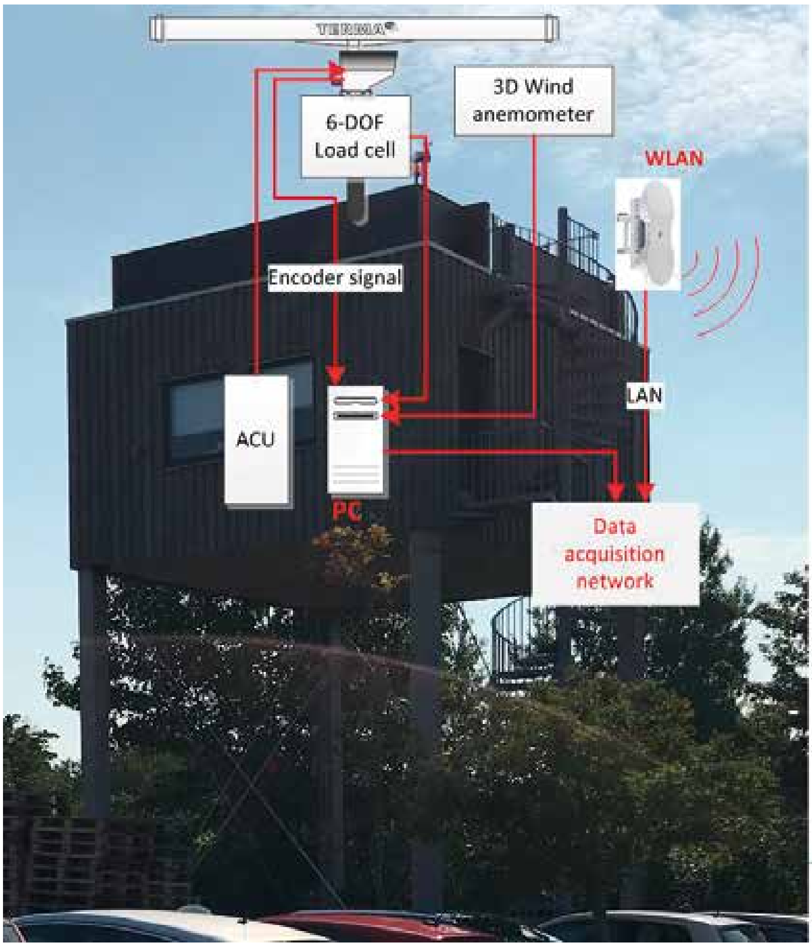

Figure 3a: Schematic of test setup. Rotating antenna and 3D wind anemometer.

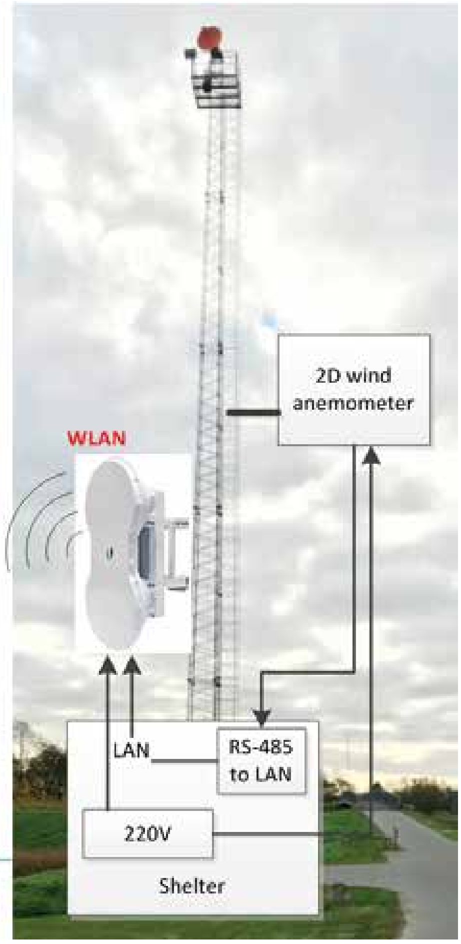

Figure 3b: Remotely placed 2D wind anemometer

The test is only short-term (3-6 months) in comparison with the typical life-time of an antenna, but during the test period, the actual loads measured on the antenna can be compared to those predicted based on the remote 2D wind anemometer readings. Since the primary interest is fatigue, the "rainflow" model is applied.

Results:

The test is currently in progress and results are not yet available.

CONCLUSION

Wind loads:

Results are pending.

Alternative materials:

Hosting ideation workshops with external partners like those described, have not previously been practiced by Terma. In general, it has proven to be very fruitful and inspirational to ideate with external partners from unlike industries, GTS and universities. In addition to generate specific results, the format of these workshops also provides a unique opportunity for employees to expand their professional network with experts both inside and outside respective fields of expertise.

VALUE / IMPACT

Of Termas yearly turnover, a significant part is related to the radar and surveillance business. Having valid models for predicting life-time wind loads spectra, and to identify adverse wind conditions is a necessity to avoid problems for customers who install radars in extreme climates. Furthermore, valid models reduce the need of overengineering and allows a future optimal use of materials, including composites, in the design of antennas.

The Fast Track infrastructure enabled cooperation between SGRE, DTU, FORCE Technology and Terma in a way that is virtually impossible in a normal customer-supplier relationship.

The potential outcome of this R&D Track is that Terma can employ new tools and enable the development of guidelines for installation of radars in adverse climatic conditions. Overall, this may lead to improved customer satisfaction.

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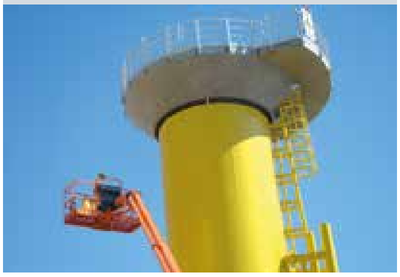
Collaborating companies:
Collaboration: Terma A/S (Terma), Siemens Gamesa Renewable Energy (SGRE), Hempel A/S (Hempel), FORCE Technology (FORCE), Danish Technological Institute (TI), Aalborg University (AAU), Technical University of Denmark (DTU)

Fast Tracks

Fast Tracks aim at meeting the immediate needs, solving a given challenge within the shortest possible period. A problem-solving model has been set up to ensure that a challenge is acted upon as fast as possible, establishing the connection between the problem owner and the right group of experts.

Fast Track 1: Crack resistance in epoxy coatings

Fast Track 2: Corrosion protection of monopiles



Fast Track 3: Wind loads

Fast Track 4: Weld beads

Fast Track 5: Strategy for corrosion sensor mapping

Fast Track 6: RoHS/REACH compatible surface treatment of aluminum

Fast Track 7: DSC and TGA as strong analysis tools for coatings

Fast Track 8: Surface hardening of cast iron

Fast Track 9: Creep characteristics of PEEK

Fast Track 10: Surface treatment of cast iron

Fast Track 11: Optimizing surface protection system of wind turbine towers

Fast Track 12: SolarSack "solar water disinfection"

Fast Track 13:
TRD wear testing

Fast Track 14: DANCOP - improving safety impact barrier

Fast Track 15: Viable substitute to Chrome-VI surfaces in food zones and optimizing of surface

Fast Track 16: Degradation of GFRC materials from wind turbine blades

Fast Track 17: Optimising material selection in connection with biodiesel production

Fast Track 19: Current collector for electrochemical cell

Fast Track 20: 3D printed valve housing – from design to verification testing

Fast Track 1: Crack resistance in epoxy coatings

SUMMARY

A demand for rapid curing coatings combined with many unknown factors from production to service life formed the basis of examining the processes of drying, curing and internal stress development of epoxy coatings. An extensive study on a fast-curing epoxy system was carried out and reliable methods to illustrate each process were identified.

OBJECTIVE / PURPOSE OF PROJECT

There is a continuous demand for rapid curing coatings with less cost and a lower content of volatile organic compounds. However, such systems have higher risk of crack formation and related mechanical coating failures.

The challenge is that the coating is exposed to many different scenarios in its lifetime; how is the coating applied and under which circumstances, is it hot, cold, humid or in the sunlight? What is the condition of the substrate? Which film thickness is obtained and is it evenly applied? And how does all this affect the crack resistance of the coating?

The aim of this fast track is to understand the interaction between drying, curing and stress development of epoxy coatings.

RESULTS & DISCUSSIONS

We investigated the processes that actually happen in the coating during drying and curing, and then exposed coated substrates to the shim bending method, to check the stress development.

Drying:

Coating was applied to clean glass substrates in 8 different thicknesses from 200 µm to 2000 µm. The weight of the samples was then measured for up to 56 days. It was observed that higher film thickness slows down the evaporation and after several weeks of drying, the thick paint films developed enough internal stress to rip the glass substrate apart.

Curing:

We studied the curing of three different coating systems using differential scanning calorimetry (DSC) instead of infra-red spectroscopy, which is typically used to monitor such reactions, as with the latter method it can be difficult to follow the last part of the reaction. Even after 50 hours we observed that curing was still happening.

Internal stress development:

We wanted to understand how much stress is actually generated in the coating without influence from environmental factors. Hardened shims of spring steel were sprayed with paint in different wet film thicknesses. Immediately after pain application the shims were transferred to at holder and the bending of shims was monitored for 48 days.

Figure 1: Bending increased with increase of dry film thickness

We gained extensive understanding of the fact that the coating is actually still changing and developing after it has been put into operation. And this is of importance for the long-term performance of the coating as we never know the exact environment of the end use.

CONCLUSION

The first steps into understanding the interplay of drying, curing and internal stress development were taken. We identified reliable methods for each process and carried out an extensive study on a fast-curing epoxy system.

It was possible to identify methods to get better understanding of both drying, curing and stress development. Drying was quantified by simple weight loss experiments, curing was followed with DSC and for understanding stresses the shim bending method was applied.

VALUE / IMPACT

Of Termas yearly turnover, a significant part is related to the radar and surveillance business. Having valid models for predicting life-time wind loads spectra, and to identify adverse wind conditions is a necessity to avoid problems for customers who install radars in extreme climates. Furthermore, valid models reduce the need of overengineering and allows a future optimal use of materials, including composites, in the design of antennas.

The Fast Track infrastructure enabled cooperation between SGRE, DTU, FORCE Technology and Terma in a way that is virtually impossible in a normal customer-supplier relationship.

The potential outcome of this R&D Track is that Terma can employ new tools and enable the development of guidelines for installation of radars in adverse climatic conditions. Overall, this may lead to improved customer satisfaction.

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Collaborating companies:
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Fast Track 2: Corrosion protection of monopiles

SUMMARY

Unexpected internal corrosion in monopile foundations calls for updated corrosion protection strategies. A literature study was carried out and recommendations for future corrosion protection of monopiles were discussed with interested parties in an attempt to obtain consensus on the procedure for future corrosion protection.

OBJECTIVE / PURPOSE OF PROJECT

By understanding the internal corrosion protection malfunctions for monopile foundations it is believed that discussions with contractors in the wind energy business may lead to a consensus in selecting a strategy with a high potential for both industrialized mass production and a low risk for failure. Hence cost will decrease, thus enabling wind power competitiveness.

RESULTS & DISCUSSIONS

Historically, internal corrosion in monopile foundations has led to unexpected costs in relation to retrofits and repairs. Years ago, it was a common belief that internal corrosion would never be an issue to consider as the monopile was assumed to be a sealed compartment. However, several monopiles have been inspected since and unexpected corrosion rates have occasionally been observed.

Figure 1: Corrosion inside offshore wind turbine foundation

Coating the inside of an existing monopiles is not realistic and the second-best solution is to retrofit CP (cathodic protection), either GACP (galvanic anode cathodic protection) or ICCP (impressed current cathodic protection). However, offshore repair is always very expensive and should be avoided on future offshore structures.

The aim of the Fast Track was therefore:

- To review and extract learning on monopile corrosion protection in order to avoid any mistakes in the future
- To give more recommendations for future corrosion protection of monopoles
- To discuss the recommendations with other interested parties in the industry – also outside the societal partnership.

The literature available today suggests that it is difficult to maintain an oxygen starved compartment of the size of a monopile. Recent guidelines even advise caution against using this approach.

Recommendations on new corrosion protection strategies were therefore given.

It was found that it is difficult to come up with a universally applicable solution for all monopile foundations as these are typically highly customized. However, this investigation opened for discussions with contractors in the wind energy business attempting to obtain consensus on future corrosion protection strategies thus eliminating repetition of previous mistakes.

CONCLUSION

It is our view that overall recommendations for future foundations should be focused on either an optimized fully closed structure or fully open structure. A redesigned closed foundation includes removal of problematic components such as inspection hatches, grout connections, and seals. The recommendations on open monopiles include coating and CP system internally as well as externally.

VALUE / IMPACT

In extension to the project the recommendations were distributed to external stakeholders within the offshore business. The results were used for discussions with contractors. On basis of the response from these the recommendations were revised to reflect common ground. The resulting document reflects the most common and shared views of the corrosion issues among the stakeholders.

The process resulted in more unification across the business and made sure that all parties were aware of the experiences within the field of internal monopile corrosion protection. Today, the results are reflected in current guidelines as well where oxygen starvation is no longer recommended for such inspectable compartments. When the strategy is fully implemented at consultancies, wind turbine producers, and subcontractors this unification for the product will result in a lower production cost due to the possibility of mass production for the subcontractors.

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March 2016 – July 2016

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Fast Track 3: Wind loads

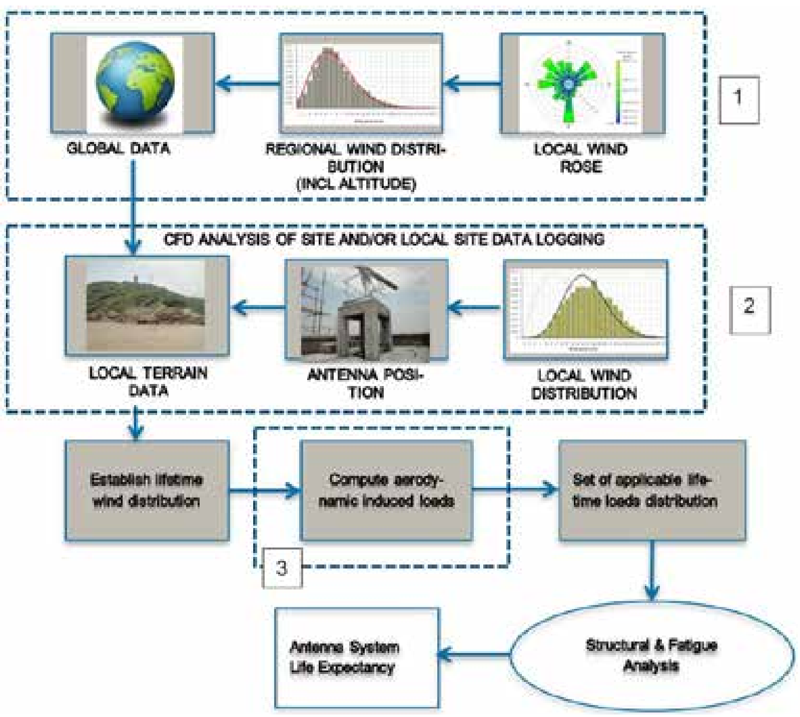
SUMMARY

The use of alternative materials and/or manufacturing processes when designing the future radar components, can be a significant factor to improving performance and manufacturing costs. To optimize use of materials and reduce risk of employing new philosophies in a design process, it is imperative that the loads set is valid for the specific application, as this will allow less conservatism.

The process of arriving at a set of life-time induced wind loads, is divided into a Global and a Local wind assessment in order to estimate the life-time wind spectrum. This is followed by a transformation of the lifetime wind spectrum into a lifetime load spectrum. The scope of Fast Track 3 has been to improve knowledge within these 3 areas.

1. Global conditions
2. Local conditions
3. Aerodynamic loading

Below diagram outlines a process for assessing the lifetime induced wind loads of an antenna, which is the required input for structural fatigue analysis and lifetime estimation.



In this Fast Track, knowledge of tools used in the wind power industry to analyze and predict wind conditions for wind turbines, is transferred to the radar business which, in some cases, experience similar challenges in determining the wind conditions for radar antennas installed in extreme environments. WAsP and WAsP Engineering (WEng) are tools developed by DTU Wind Energy for wind resource assessment and wind conditions for fatigue loads (ref. http://www.vindenergi.dtu.dk/english/software). These look very promising for the analysis of both radar site suitability studies at early stages of radar projects and also for adding strong technical support to existing customers experiencing mechanical performance difficulties at sites with extreme terrain and climatic conditions.
With respect to the determination of localized wind conditions, the overall conclusion is, that the wind anemometers installed on antenna sites can be significantly biased for determining the correct localized wind spectrum. Hence, using single point wind anemometers alone to measure the wind conditions must be considered wisely.

The full-scale wind tunnel test of a rotating radar antenna showed overall good correlation between models and test data. Some discrepancies were identified but believed to be related to the test setup rather than the errors in the models.

OBJECTIVE / PURPOSE OF PROJECT

The objective of Fast Track 3 is to:

1. Investigate if tools from the wind power industry can be used to estimate wind conditions for radar sites.

2. Conduct wind tunnel test to validate wind loads models for a radar antenna.

RESULTS & DISCUSSION

Global conditions

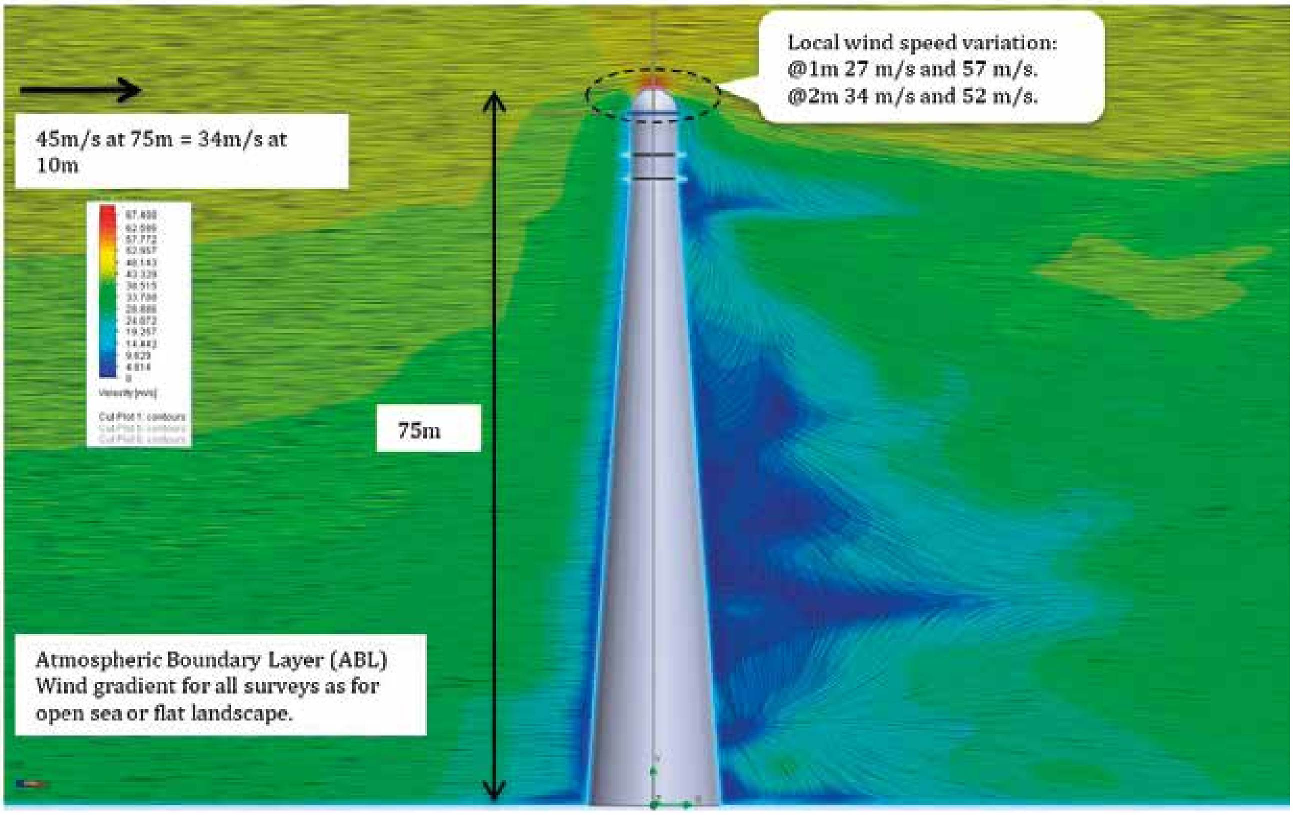
The investigation of Global Wind Conditions started with a meeting at Siemens Wind Power in Brande on May 27, 2016. At the meeting, Per Hessellund Laursen (Head of research at Siemens) gave an introduction to the background for wind turbine certification at Siemens.

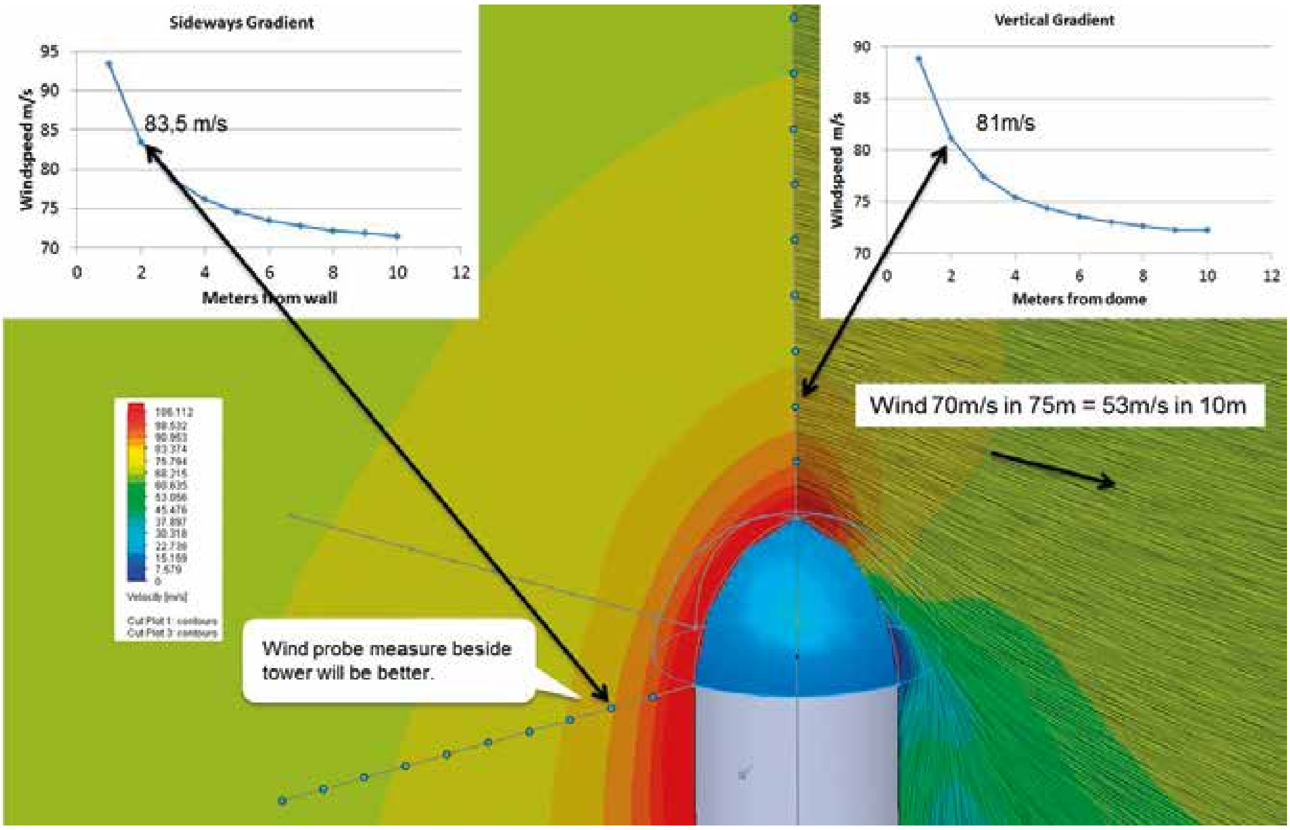
During the meeting at Siemens, the possibility of using the software WAsP for the prediction of wind distribution was discussed. Developed by DTU Wind and being the industry standard for more than 15 years, WAsP helps predicting the annual energy production for wind turbine farms around the globe as well as extreme wind occurrence (50-year). WAsP gives the user the opportunity to use sampled data or wind atlas mesoscale data from several international organizations and institutions, for predicting mircoscale wind distribution and thereby associated energy production and extreme winds. Particularly, the intermediate wind distribution data and extreme wind prediction is interesting for Terma, in terms of estimating the antenna lifetime.

As a part of Fast Track 3, Terma purchased a license for the use of WAsP, and conducted a trial experiment. A case involving an antenna site was investigated. This underlined the effectiveness of WAsP in calculating local wind speeds that otherwise would be unobtainable, by only looking at nearby weather stations.
WAsP & WEng look very promising for the analysis of both radar site suitability studies at early stages of radar projects and also for adding strong technical support to existing customers experiencing mechanical performance difficulties at sites with extreme terrain and climatic conditions.

Local wind conditions:

The investigation of Local Wind Conditions was based on Computational Fluid Dynamics (CFD) studies of simplified building structures. The localized wind effects were investigated in terms of how much the wind was accelerated compared to the far-away wind field, and how much the wind vector was angled compared to horizontal. In coastal regions existing structures such as lighthouses provide an attractive platform for installation of coastal surveillance radars. As shown in the example below, the wind around the top of the light house varies significantly, depending on the position of measurement. Compared to far field wind conditions, the local wind speed can deviate 20-50%, depending on the distance to the structure. Normally, installation of radar antennas would be in this localized area experiencing high velocity gradients and incident angles. Furthermore, the size of radar antennas installed would be subjected to significantly skewed winds, resulting in increased loading.





Similar conclusions are made for larger rectangular shaped buildings. For these structures, large variations between the horizontal and vertical components are also found.

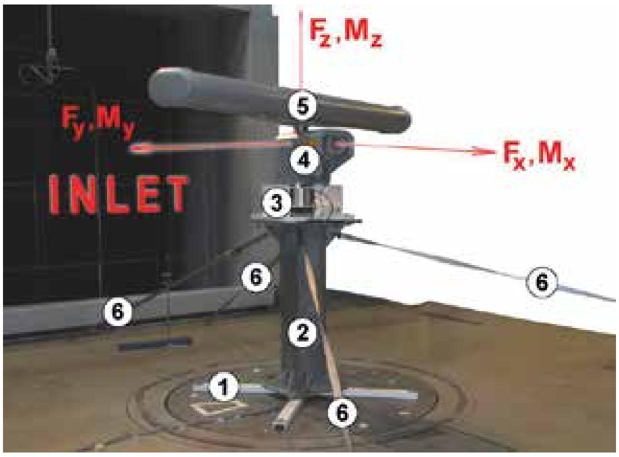
The overall conclusion is, that the wind anemometers installed on antenna sites can be significantly biased by the local structure, for determining the correct localized wind spectrum.

Aerodynamic loading

Over several years, Terma has developed models for calculating wind loads on antennas and performed a number of tests and experiments to verify and validate drag and lift forces on antenna profiles. Dynamic models of full-scale rotating antennas are based on the blade element methodology historically used in the wind power industry. However, a validation of the integral reaction forces and moments on a full-scale rotating antenna, operating at a Reynolds number range similar to actual installations, still remained.

The objective of this activity was to provide test data of loads acting on a rotating antenna for validation of Terma's standard calculation methodology, for ideal wind fields. With help from FORCE Technology in Lyngby, a wind tunnel test of a rotating antenna was performed in Velux' wind tunnel in Østbirk. This is the largest wind tunnel in Denmark that can approach wind speeds of up to 45m/s. Despite this, size is still the limiting factor. The largest antenna profile that could fit in the wind tunnel was a 9' compact antenna that was specially constructed from existing parts. This antenna is of the same size as the smallest antennas developed by Terma. However, this was still sufficient for validation of the models.

The antenna and turning unit placed in the wind tunnel, on a 6 degree of freedom (DOF) load transducer, as shown below. The test is performed at different angles of inclination (attack) by tilting the antenna platform.

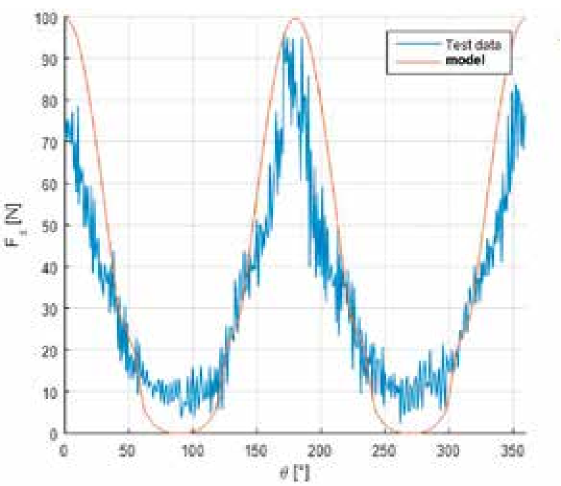


The different test components are labelled as follows:

1. *Foundation*
2. *Piedestal*
3. *Tilt mechanism, load transducer*
4. *TU*
5. *Antenna*
6. *Lashings*

To generate reaction loads as a function of angular position of the antenna, all 6 DOF loads and the turning unit built-in resolver signal re sampled simultaneously at 1kHz. FORCE Technology in Lyngby, who possesses a vast experience with wind tunnel instrumentation, performed the instrumentation.

Generally, it was found that the computational model correlates well with the experimental data. As an example, the figure below shows the correlation between test data and the model, for horizontal forces (Fx), for both raw and filtered data.



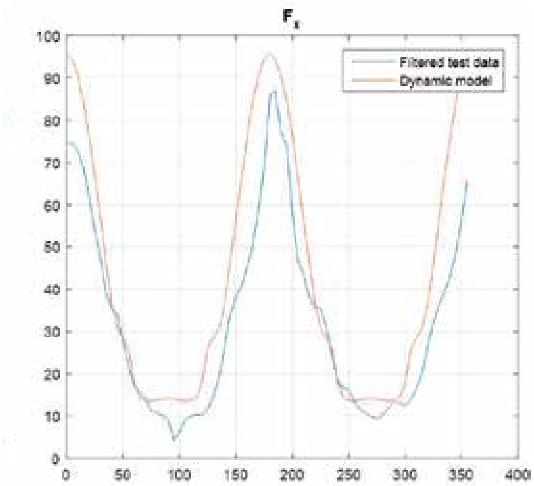


Figure: Horizontal force (Fx)

Based on the test results, it was concluded that the models used to predict aerodynamic induced wind loads are valid for ideal wind conditions.

CONCLUSION

WAsP & WEng look very promising for the analysis of both radar site suitability studies at early stages of radar projects and also for adding strong technical support to existing customers experiencing mechanical performance difficulties at sites with extreme terrain and climatic conditions.

The overall conclusion is, that the wind anemometers installed on antenna sites can be significantly biased by the local structure, for determining the correct localized wind spectrum, and therefore must be installed wisely for their purpose.

Based on the wind tunnel test results, it was concluded that the models used to predict aerodynamic induced wind loads are valid for ideal wind conditions produced in a wind tunnel.

The outcome of Fast Track 3 is that Terma applied for continuing the work as an R&D track in 2017 and 2018. The R&D track shall continue the investigations of the model validation and conduct full-scale long-term measurements on a full-scale antenna installed operating in the field. This shall lead to a validation of the complete analysis methodology from Global Wind Distribution, through Local Wind Distribution to the arrival of lifetime induced wind loads. In parallel with this, workshops will be set up with the partners of the Fast Track, with the purpose of discussing future use of alternative materials and fabrication methods.

VALUE / IMPACT

Of Termas yearly turnover, a significant part is related to the radar and surveillance business. Having valid models for predicting life-time wind loads spectra, and to identify adverse wind conditions is a necessity to avoid problems for customers who install radars in extreme climates. Furthermore, valid models reduce the need of overengineering and allows a future optimal use of materials, including composites, in the design of antennas.

The Fast Track infrastructure enabled cooperation between SGRE, DTU, FORCE Technology and Terma in a way that is not practical in a normal customer-supplier relationship.

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Collaborating companies:
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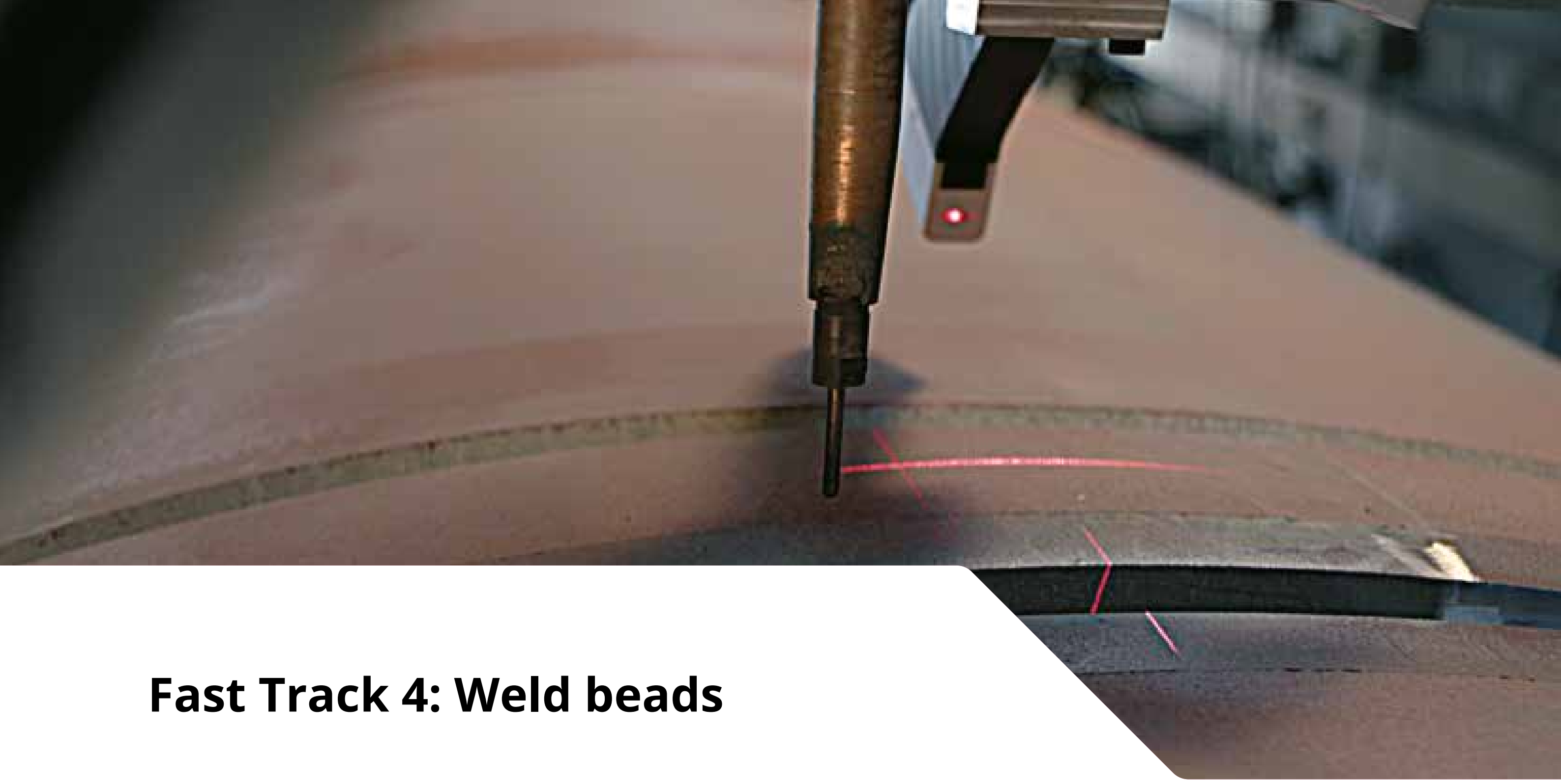

Fast Track 4: Weld beads

SUMMARY

A preliminary study of the influence of the weld bead on the S-N properties (fatigue properties) has led to the belief that monopiles and towers can be designed with modified S-N curves thus leading to massive steel savings. The Fast Track elaborated on the potential savings and resulted in an application for a project extension.

A JIP, Joint Industry Project in corporation with partners from Vattenfall, Ørsted, Cowi, Rambøll, SGRE and DTU was later granted partially on basis of what was generated and launched during this Fast Track.

OBJECTIVE / PURPOSE OF PROJECT

The Danish Wind Industry Association has prior to this project established three workgroups. The focus of these is to reduce the costs associated with the production of various elements in wind turbines. One group has focused on the support structures, including both towers and offshore foundations. For the latter the focus has primarily been on monopiles. The group has identified weld-beads, as well as removal or optimization of surface texture on these, as important parameters in a cost-optimized design of such structures. The hypothesis is that a more detailed understanding of how the surface morphology impacts the fatigue properties, will lead to significant advantages when selecting design for offshore structures. The project will seek to improve the understanding to optimize the design basis.

The project therefore focusses on a detailed clarification of the effect of weld beads removal during tower and monopile manufacturing. For both, it is desired to investigate if:

1. More accurate S/N-curves can be obtained for different removal processes in order to create a better design basis.

2. The currently applied weld-bead removal processes can be simplified, substituted or eliminated, without compromising the end-result with respect to fatigue properties.

RESULTS & DISCUSSIONS

The project is a continuation of an already-initiated project, SLIC, which was a study on how the removal of potential crack initiators impacts the propagation of the cracks. SLIC did not focus on the importance of the surface texture after removal of the weld bead. Likewise, it was not a study of various low-cost automated processes suitable for removal of these weld-beads.

Moreover, investigations showed that surface treatment of weld beads has a large impact on the obtained fatigue properties. The project confirmed that it is feasible that machining alone, leads directly to significantly less conservative S/N-curves, than those used for non-treated welds suggested by the guidelines. With introduction of novel automated removal processes for weld beads, it is increasingly important to clarify how the surface morphologies impact the fatigue strength of the welds and hence how the processes impact the applied design curves.

CONCLUSION

The project resulted in an application for a EUDP project. The project has later been granted as "WindWeld". Partners are Vattenfall, Ørsted, Cowi, Rambøll, SGRE and DTU.

VALUE / IMPACT

The proposed project aimed to contribute with cost reductions in relation to offshore wind structures. With reduced cost of sustainable energy technologies, these will become even more cost competitive, thus eliminating the need of fossil resources such as oil and gas. The project created on basis of this Fast Track is expected to bring cost benefits across the entire Danish wind industry with an expected impact on LCoE (Levelized Cost of Energy) up to -0.5%. The weld bead removal technique is expected to bring continuous production of e.g. towers in Denmark.

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December 2016 – February 2017

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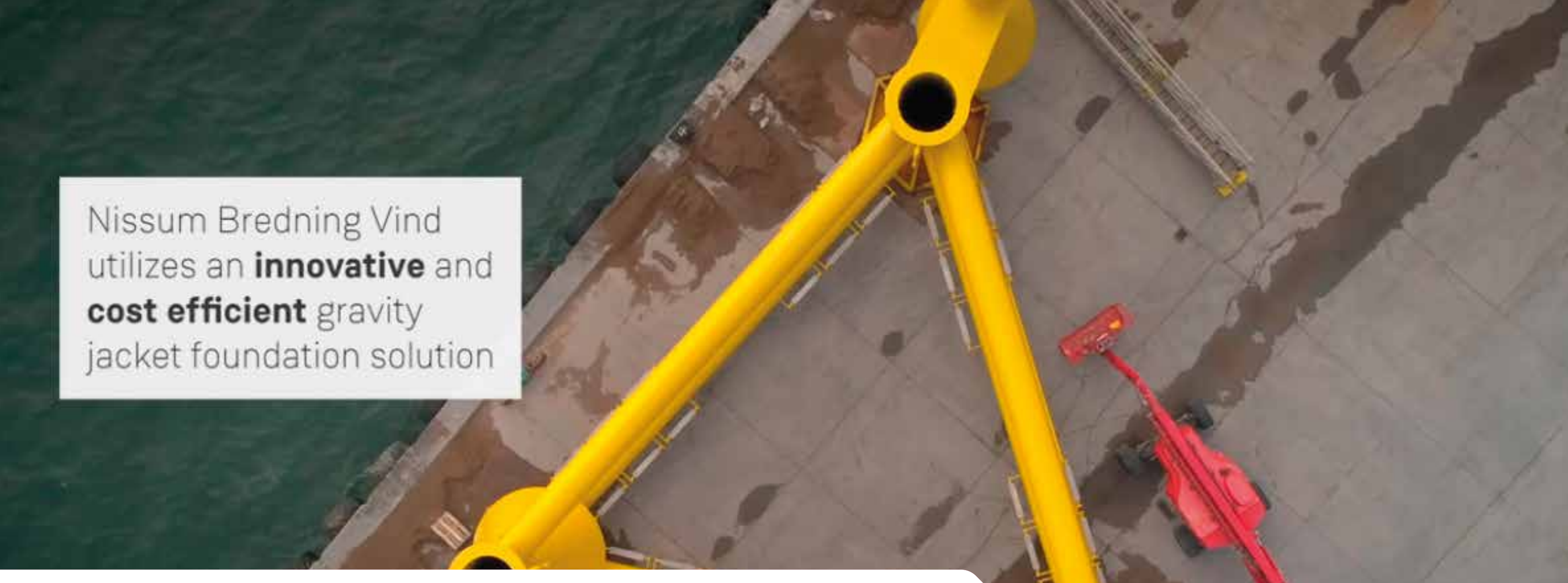

Fast Track 5: Strategy for corrosion sensor mapping

SUMMARY

The Fast Track reviewed available as well as future techniques for condition monitoring of the integrity of offshore structures. The aim was a strategy proposal and means for monitoring, hereby securing a minimum amount of maintenance to the offshore substructures. The Fast Track served as a brick in the initiation of a shift from schedule-based maintenance to condition-based monitoring, thus eliminating the need for costly inspections, maintenance and retrofit solutions.

A strategy for monitoring was therefore proposed on basis of the FastTrack findings. The literature survey presented in the reports was generic, while the proposed monitoring strategy was somewhat tailored for the Nissum Bredning "demonstration" project.

OBJECTIVE / PURPOSE OF PROJECT

For future offshore projects SGRE will potentially have the responsibility for service and monitoring of offshore foundations including both monopiles and offshore jackets. With the previously launched Fast Tracks on corrosion protection of monopiles, as well as in the slipstream of an EUDP project, this Fast Track aimed to clarify the options for monitoring.

By now SGRE has a long tradition within monitoring with the TCM system (Turbine Conditions Monitoring). Parameters such as the vibration level are monitored at selected locations throughout the turbines. Such information is used to remotely predict or even detect immediate failures. On top of the TCM system other monitoring systems constantly check the situation in the turbine including temperature, noise, frequency etc. In the future the range of sensors will increase, and even more parameters will be monitored, including corrosion rates, condition of CP (cathodic protection) etc. SGREs knowledge within monitoring of the structural integrity can therefore with small adjustments and amendments also be extended to cover the structural integrity of the foundation.

RESULTS & DISCUSSIONS

In summary the project included:

- Short state-of-the-art literature review of typically supplied corrosion sensors – both for offshore corrosion monitoring and other areas. Concepts such as the traditionally used coupons have not been evaluated since they are unsuitable for maintenance-free offshore structure and hence remote data acquisition. All proposed sensors should be suitable for integration into the TCM system thus allowing continuous and remote monitoring and thereby enabling condition-based maintenance.
- Discussion of hot-spots on jacket type constructions.
- Summary of state-of-the-art sensors suitable for monitoring of the Nissum Bredning jacket.
- Recommendations on where to focus on implementing corrosion- and corrosion-protection monitoring on SGREs future foundations.

Corrosion protection of subsea installations has so far primarily relied on corrosion allowance, coating and cathodic protection. Monitoring of the corrosion situation has not always been prioritized since inspection of offshore installations has been a natural part of the offshore business. This is, however, likely to change as turbines move to even deeper and more remote locations. It is therefore increasingly important to rethink corrosion protection and monitoring strategies for offshore turbine installations. This can, among other things, be done by:

a) Redesign of the structures with focus on previous learnings and failures in relation to corrosion. Avoid repetition of expensive mistakes.

b) Design that ensure a lifetime of >25 years. This requires new focus on coating qualification and specification.

c) Installing sensors for condition-based monitoring.

Examples are:

- Implement real time surveillance of the corrosion-protection state allowing to act in time or to evaluate remaining lifetime of e.g. coatings and anodes
- Increase robustness of CP systems
- Improve modelling and prediction of protective potentials
- Monitoring of the corrosion state allowing condition-based maintenance
- Create low-cost and standardized corrosion protection solutions suitable for a novel mass-produced product portfolio.

CONCLUSION

With the application of well proven sensors it is possible to propose a monitoring strategy which can supply owners with valuable and real time data, suitable for both active and passive condition monitoring at remote locations of the entire turbine installation. Based on the review it was found that advanced techniques are not necessarily required for obtaining a realistic view of the structures overall.

The recommendations were given on basis of well proven principles which are found adequate for future monitoring. More detailed discussions should, however, again always be initiated on detailed design briefs on a case to case basis.

The recommendations for universally applicable sensor types to be included in future corrosion protection strategies are in a prioritized order;

1. Potentials monitoring through commercially available reference electrodes
2. Electrical Resistivity sensors (different types for different areas – some are commercially available).

VALUE / IMPACT

The proposed project will contribute with cost reductions in relation to offshore wind structures. With reduced cost of sustainable energy technologies, these will become even more cost competitive, thus eliminating the need of fossil resources.

With the implementation of this corrosion monitoring the economic analysis estimates a cost reduction of potentially 121.500-421.500 DKK per offshore turbine.

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Duration of project:
December 2016 – February 2017

Collaborating companies:
Siemens Gamesa Renewable Energy A/S (SGRE), FORCE Technology and Danish Technological Institute (TI)

Fast Track 6: RoHS/REACH compatible surface treatment of aluminum

SUMMARY

In this project a literature search and neutral Salt Spray (NSS) testing has been performed on four typical aluminum alloys used in Terma products and three different REACH compliant chemical conversion coatings plus Alodine 1200S as a non-REACH compliant reference coating.

A preferred candidate has been identified as replacement for the hexavalent chromium-based coating.

The project has identified possible problems with adhesion of REACH-compliant primers to the REACH-compliant coatings, a subject which will be handled in future internal projects.

OBJECTIVE / PURPOSE OF PROJECT

Chemical conversion coating (also called passivation) and anodizing based on chromates with hexavalent chromium perform excellent and are used extensively in civil and military applications. The EU REACH-directive bans these substances - alternatives have existed for many years, but test results are not unambiguous, test alloys are limited to a few standard materials, and the processes subject to test are not all available in Europe.

Furthermore, Terma has a need to replace a non-REACH compliant primer for aerospace parts.

RESULTS & DISCUSSIONS

The work has been performed mainly by DTU and Terma, but in the initiating phases with contribution also from TI and discussions with FORCE and Siemens.

The work has included:

- Extensive literature search of existing test results, comparison of results and extract of learnings
- Characterization of alloys with respect to corrosion sensitivity
- Survey of available surface treatment and candidates for test
- Test plan and involvement of suppliers for test panels and surface treatment
- The project has been expanded to include a REACH-compliant primer for aerospace products, including corrosion test and adhesion to typical aluminum alloys.

DTU has been the main contributor in performing the new surface treatments and to conduct salt spray test, see Figure 1. The first results of salt spray test are different than expected, especially when we compare “old” and “new” processes. Retesting, including investigation of a couple of test parameters has been planned and will hopefully result in some very useful learnings for future test.

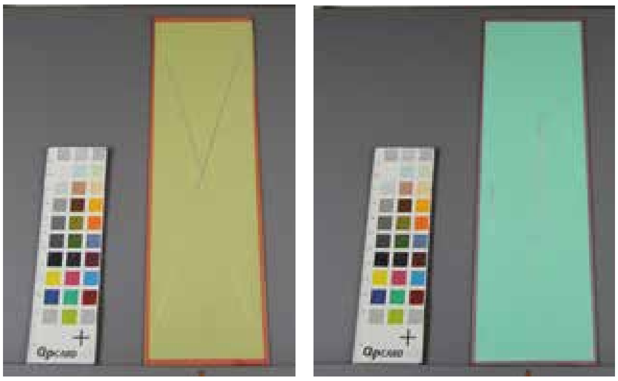
The work has been concentrated on investigating REACH-compliant passivation processes for aluminum, i.e. trivalent chrome based and chrome-free chemical conversion coatings.

Figure 1: NSS test setup at DTU

Corrosion and paint adhesion test have shown significant differences between the processes and significant differences in results on different alloys, more than normally seen with the well-known processes, that have been used previously, see Figure 2 and Figure 3. With respect to chemical conversion coating, only the trivalent chromate-based coatings can be approved for use at Terma, and of the coatings tested, only the process from SurTec can be approved.

From the testing we have learned, that:

- The process shall be adapted to the aluminum alloy
- The process shall be adapted to either maximum corrosion protection or to pre-treatment before paint



Left: Figure 2 "Old primer" Right: Figure 3 "New primer on chrome-free passivation", sample with corrosion pits

Only one specific manufacturer's process has been approved for general use (SurTec 650), and, until now, only for alloys EN AW-5754, EN AW-6061 and EN AW-6082.

More testing on alloys AL-2024 and Al-7075 has been performed together with SurTec as an internal project at Terma. The evaluation of the test results will be performed during March 2019.

CONCLUSION

The REACH compliant chemical conversion coating identified as replacement for the hexavalent chromium-based coating is SurTec 650.

Conclusion with respect to a REACH compliant primer for aerospace application is not clear – more testing on different alloys and pre-treatments must be performed, but we will do this in internal projects.

VALUE / IMPACT

The Fast Track project on RoHS/REACH compliant surface treatments for aluminum has provided us with new insight in aluminum alloys and the challenges related to surface treatment of these alloys.

Supported by literature search we have identified, the chemical conversion process that we will introduce on future products, but we have also learned about the challenges with the new process and how to address them.

In addition to this, the project has introduced us to a network of companies and knowledge centers, that has inspired us to a current internal task of writing a handbook for designers, including categorization of corrosion environment and selection of suitable surface treatments on metallic surfaces.

We will continuously improve our design solutions and expect to use the network as inspiration and as a "review team".

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Collaborating companies:
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Fast Track 7: DSC and TGA as strong analysis tools for coatings

SUMMARY

Modern coatings are complicated systems with many components and it is thus difficult to interpret the DSC (Differential Scanning calorimetry) data. Nevertheless, our initial studies show that the technique is very promising for understanding, among others, drying and curing and endothermic relaxation. In addition to the outlined DSC-study we have conducted thermogravimetric analysis (TGA) of the same samples. This has given additional information that helped us understand how the coating is influenced by thermal energy.

OBJECTIVE / PURPOSE OF PROJECT

The purpose of this Fast Track is to explore two most commonly used analytical techniques (DSC and TGA) to investigate how curing, drying, endothermic relaxation and glass transition temperature affects the final viscoelastic properties of a coating.

RESULTS & DISCUSSIONS

The work done in the Fast Track on Crack resistance in epoxy coatings has among others included a screening of the use of Differential Scanning calorimetry (DSC) and thermogravimetric analysis (TGA) as a tool for thorough coating analysis. It can help us understand how the interplay between curing and solvent evaporation works and also how the coating is influenced by e.g. weathering.

The aim of this project was to quantify the amount of curing and solvent entrapped into the paint film which in turns give rise to the elastic and plastic properties. The domination of plastic properties due to the presence of solvents and uncured polymer lead to develop domains of lower and higher stresses within the partially dried paint films. This imbalance in elastic and plastic properties results in the coating failure. This knowledge helps us create a nice balance between elastic and plastic properties.

Differential Scanning calorimetry (DSC) is used to study curing process with time and temperature. Figure 1 shows curing profile of an epoxy coating with respect to time and the results indicate that as the curing proceeds the curves become flatter and flatter.

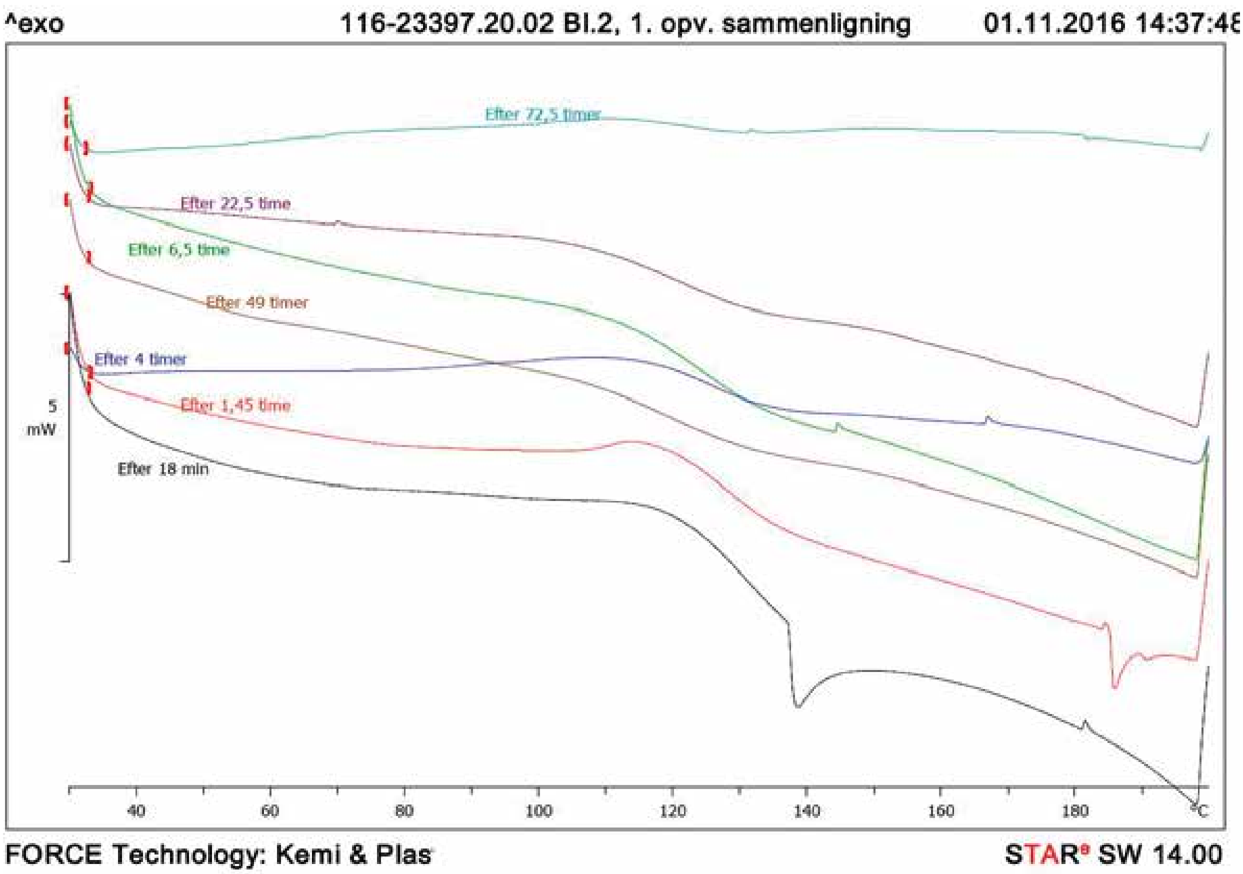
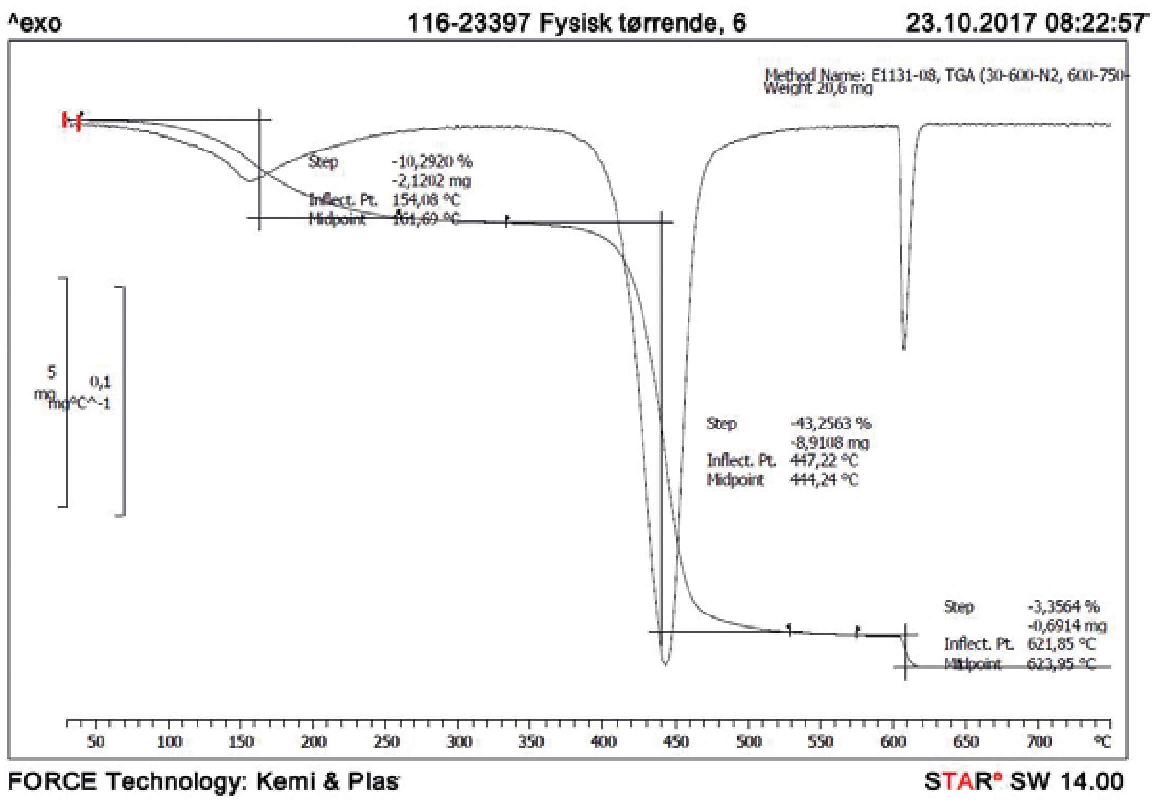


Figure 1: DSC graph

Thermogravimetric analysis (TGA) is used to investigate the solvent retention in the paint film.

The graph in figure 2 shows that 10 weight percent of the total weight of paint film constitute of solvent retained into the paint film.



CONCLUSION

In this Fast Track we have made a time dependent investigation of the behavior of 3 epoxy coatings as well as solvent retention with the collaboration of FORCE Technology. The signatures from the DSC showed the development of glass transition temperature (Tg) during epoxy cure and the footprints from the TGA provided the information of solvent retention and its effects on the Tg. The epoxy coatings started to develop Tg after 3 hours and a slow increase in Tg was observed during the entire curing profile (65 hours). The amount of solvent decreased fast (evaporation) at the start and became slow after 3 hours. Approximately 5-10 % of the solvent was retained into the partially cured coating after 24 hours, this was attained by TGA. The results showed that DSC and TGA are strong analysis tools to investigate the curing process with time. Further studies are required to understand how different coatings respond at different temperatures, humidity and exposure to salt spray.

VALUE / IMPACT

Solutions and knowledge generated in this Fast Track have been implemented in the R&D Track C ”Development of new C5 coatings ”. Once we have the methodology fully developed, it will be able to correlate internal stress and curing speed. This will be used to minimize the risk of cracking in fast curing epoxy systems (e.g. winter versions for cold climates) and reduce the number and cost of cracking and delamination complaints. The cost of these complaints is between 1 and 1.5 million euros annually.

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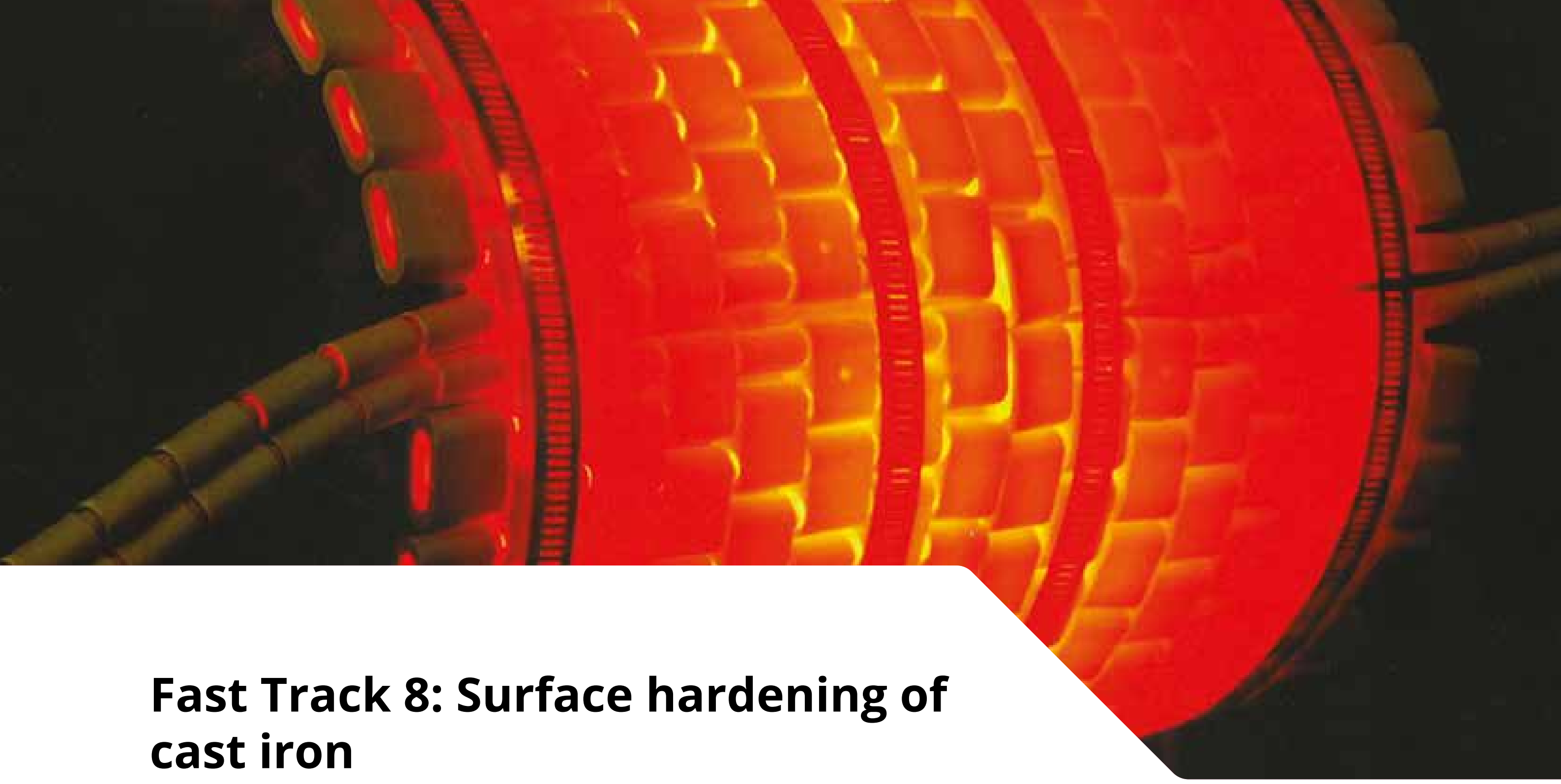


Fast Track 8: Surface hardening of cast iron

SUMMARY

In order to reduce wear on surface due to sliding (e.g. by sealings) there is a need of increasing surface hardness of ferritic ductile cast iron without changing the material in the core of the material. In this project the possibility of heat treatment by induction hardening was investigated, both by experimental work and by simulation tools. The results show that by use of a preheating step it is possible to increase the surface hardness without changing the microstructure in the core.

OBJECTIVE / PURPOSE OF PROJECT

Overall purpose of project was the question if ferritic ductile cast iron can be surface hardened, leaving the core microstructure unchanged. This will require:

- Development of a thermal process that allows diffusion of carbon from graphite nodules into the matrix (only for the surface and near surface material)
- Development of a hardening process, including quenching
- Performing microstructural investigations and hardness measurements.

RESULTS & DISCUSSIONS

The casting grade normally used in wind turbines is ferritic ductile cast iron (EN-GJS400-18LT). This is a very ductile material, but also relatively soft material, which gives risk of wear, if sliding occurs on the surface. The purpose of the project was to investigate if the surface could be hardened without changing the properties in the core of the material. It was decided to limit this project to only investigate this process which is based on heat treatment. Other process possibilities, which include partial melting (e.g. coatings), were to be investigated in another project. The main part of the work in this project was done as part of a master project at DTU.

The project contained both some experimental work and some simulation work. The experimental part of the work contained 4 steps.
Step 1 was heat treatment of small samples (2x2 mm) at different temperatures to see how carbon diffused from graphite nodules into matrix. The parts were kept in furnace for 2 minutes before they were quenched. Examples of resulting microstructure are shown in Figure 1. This experiment showed that it was possible to diffuse carbon into matrix within reasonable time.

Step 2 was Jominy end quench test for testing the hardenability of the material. The hardening depth was 1.5 to 2 mm, which was found acceptable.
Step 3 was induction heating of Ø25mm bars (see Figure 2). The samples were preheated to either 860°C or 900°C for diffusion of carbon, cooled down, heated to 860°C and quenched. This gave an increased surface hardness, but the microstructure in the core was also changed. This is a consequence of the size of samples, which was so small that it was not possible to heat the surface without also heating the center

of the sample and changing the microstructure. For larger samples as real casted wind turbine component this would not be the case. Thereby there is no conflict with the main objective of the sample (see below). Step 4 was induction heating of a Ø300 mm sample (see Figure 3). This test sample size was more representative for the size of large wind turbine castings. The process consisted of a preheating stage, cooling, heating for hardening and quenching. The surface hardness was increased up to 450HV and 10 mm below the surface the microstructure was not affected by the process.

Along with the experimental work, simulation was done by use of ThermoCalc (calculation of phase diagram) + DICTRA (simulation of diffusion controlled phase transformation) on carbon diffusion distance as function of preheating time. Results of calculated carbon diffusion distance is shown in Figure 4. Further, the distribution of heat from the induction heating process was simulated by use of COMSOL (a software for multiphysic simulations, e.g. simulation of temperature during induction heating).

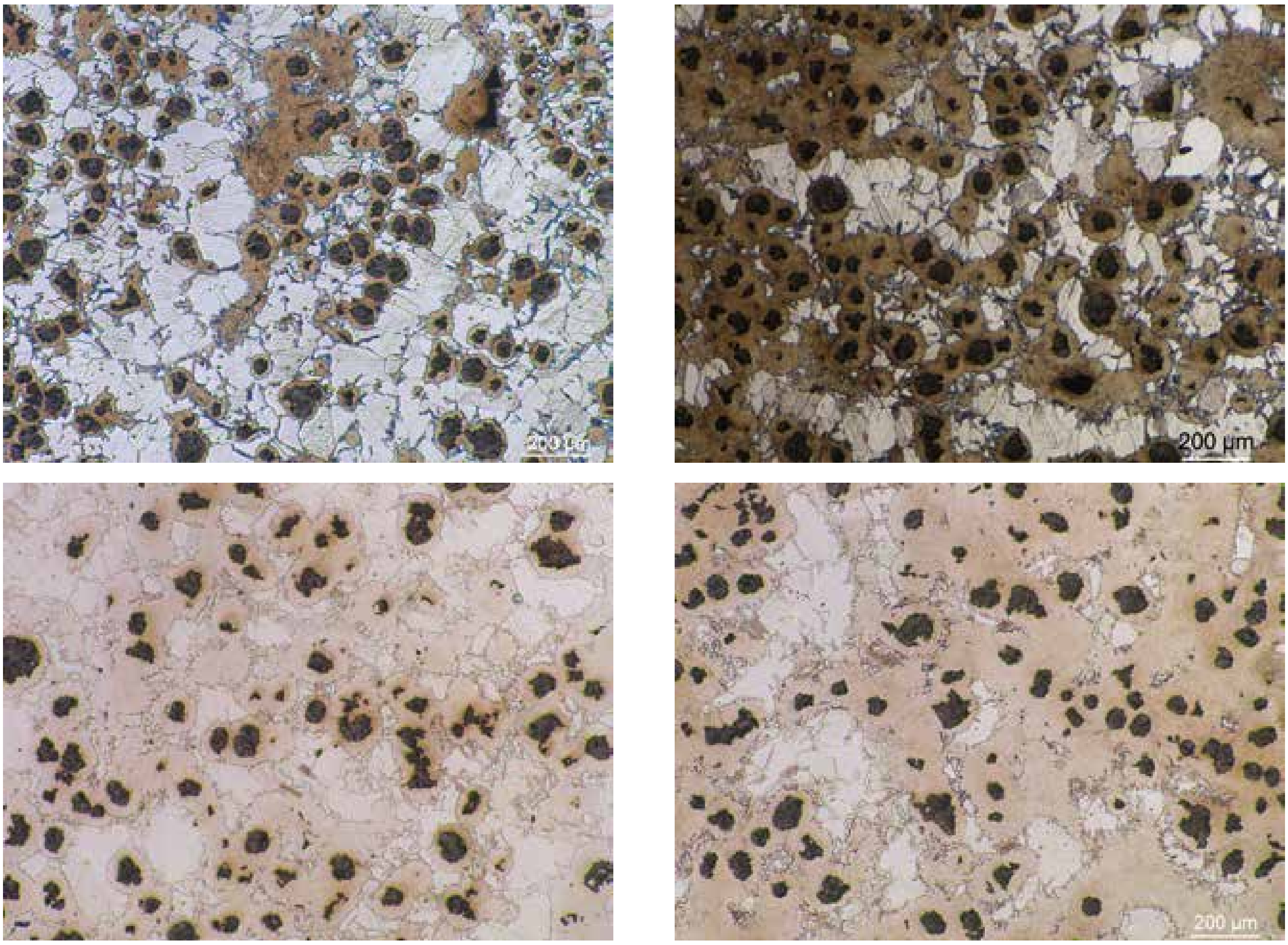


Figure 1: Heat treatment at 800, 830, 860 and 890°C, to see how carbon diffused from graphite nodules into matrix

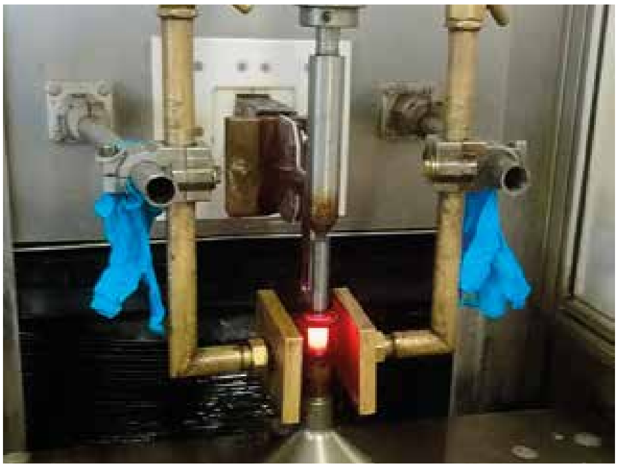
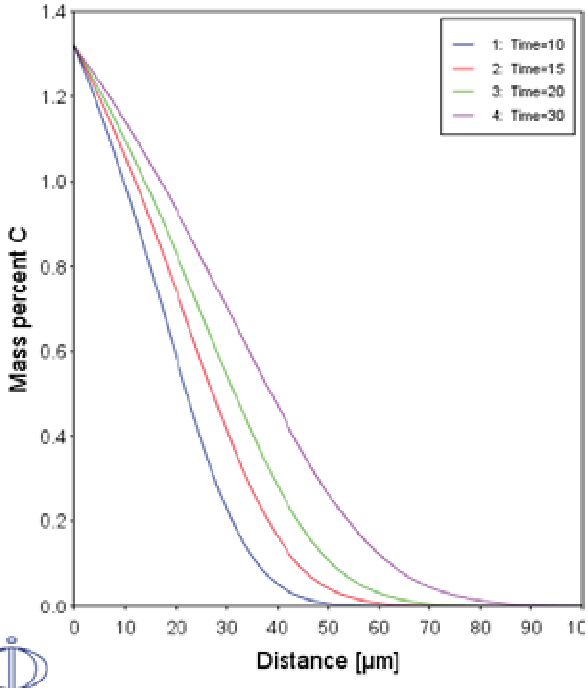


Figure 2: Induction heating of Ø25 mm sample

Figure 3: Induction heating of Ø300 mm test sample.



CONCLUSION

The main conclusion from the project was:

- Induction hardening is a feasible method to surface harden the investigated material with possibilities to upscale.
- Induction pre-heating allows for a compositional modification and increases the hardenability of ferritic ductile cast iron.
- Complimentary use of ThermoCalc + DICTRA and COMSOL was demonstrated. It can be used as an optimization tool.

VALUE / IMPACT

Value for company:

- Understanding of possibilities and restrictions of the method
- Knowledge may also to be used for evaluation of alternative thermal solutions
- Further process development in cooperation with supplier will be needed if the solution is going to be introduced on full scale sample.

Value for society:

- Relevant project for a master student
- Master student continued at Ph.D. project in co-operation with SGRE
- More powerful wind turbines.

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Duration of project:
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Collaborating companies:
Siemens Gamesa Renewable Energy A/S (SGRE) and Technical University of Denmark (DTU)

Fast Track 9: Creep characteristics of PEEK

SUMMARY

SGRE has previously performed various tests on a PEEK polymer. During this work short-term creep tests indicated a severe risk of long-term creep behavior. To mitigate this risk a Fast Track was initiated. The focus of the Fast Track was to create a model which could accurately predict the long-term creep behavior of the relevant PEEK polymer.

OBJECTIVE / PURPOSE OF PROJECT

The aim of the project was twofold;

1. From short term tests under different strain rates and temperatures the objective was to develop a model predicting the long-term behavior. The aim of this first part was to achieve a model and data for a commercial PEEK material. The results from this work can be published.

2. The same is done for a SGRE specific material. Necessary corrections are applied to the models to reflect difference in material properties. The results from this will not be published and will be available only for SGRE.

SGRE previously performed short-term creep tests under constrained compression. AAU previously conducted short-term creep tests under tension. During the Fast Track AAU performed short-term creep tests under uniaxial tension at various temperatures and stresses. AAU both performed tests and developed a model for prediction of observations in medium-term uniaxial creep. AAU compared the results of numerical simulation for medium-term creep tests with the experimental observations thereby validating the proposed model. Based on the results necessary corrections were applied to the model.

Tensile specimen after elongation and rupture.

RESULTS & DISCUSSIONS

Relevant PEEK specimens underwent testing (strain rates, relaxation and creep). Testing at elevated temperatures was also made. Initial results showed a low sensitivity to strain rate so testing in a wider range of temperatures was necessary for creating the model. Creep loads at low temperatures and high stress all resulted in premature failure.

Due to the high stiffness and low deformation to fracture and the need for testing at high temperatures, special pneumatic grips have been ordered for easy stress-free mounting. The delivery time of the grips caused a delay to the Fast Track.

CONCLUSION

The validated model is to be delivered ultimo April 2019 with a delay due to delivery time of grips.

VALUE / IMPACT

The validated model is to be used for prediction of creep behavior thus enabling successful design of the component. Having a sufficient data basis will enable more accurate design and thus prevent unforeseen costs in relation to retrofitting and maintenance of the components in question and thereby reducing the LCoE, Levelized Cost of Energy.

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Duration of project:
May 2018 – March 2019

Collaborating companies:
Siemens Gamesa Renewable Energy A/S (SGRE) and Aalborg University (AAU)

Fast Track 10: Surface treatment of cast iron

SUMMARY

The Fast Track identified surface treatment solutions for a specific type of cast-iron intended for application in one of SGRE´s products. Different requirements for the surface coating were identified early in the Fast Track. Based on these requirements different coating solutions were proposed by the partners and coated onto samples relevant for further characterization in SGRE labs. A few candidates were subsequently selected for more thorough analysis in different test rigs.

The Fast Track activities therefore covered:

- Idea generation from the partners for new possible surface treatment solutions
- Creating test samples for testing and characterization
- Characterization of the created surface treatment solutions on the cast iron substrate.

OBJECTIVE / PURPOSE OF PROJECT

TThe objective of the project was to identify a surface treatment method that creates a hard, durable and machinable surface. Good tribological properties will for this specific application be an advantage. The process chosen for the coating application might ultimately be applied to large sized components. The processes proposed therefore had to take these constrains into considerations.

During the Fast Track a workshop was setup between the partners. During the one-day workshop several brain storming activities identified solutions suitable for the application on the final component. Among the interesting surface treatment solutions were:

- Laser cladding
- Thermal spraying
- FeC coating
- Electroplated solutions
- Autocatalytic solutions.

Some of these solutions were subsequently ordered and the samples were analyzed in the lab on small sized samples as shown in Figure 1 below.

Figure 1: Test samples to be analyzed in the lab

A few candidates were later ordered in larger sizes to conduct additional tests within wear, delamination etc.

RESULTS & DISCUSSIONS

From the analysis it was found that certain candidates can secure a sufficiently robust coating on top of the cast iron specimens. In this way it was possible both to utilize the relevant cast iron grade and reduce the risk of coating failure.

VALUE / IMPACT

The chosen coating significantly increases surface properties for the relevant cast iron. Thus, allowing the material to be used in an application where it otherwise would be unsuitable.

CONCLUSION

A few candidates were identified during the Fast Track. Some were promising for the relevant application. Characterization and test of these are still ongoing and the coatings are being upscaled. Samples are being ordered.

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Duration of project:
August 2018 – January 2019

Collaborating companies:
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Fast Track 11: Optimizing surface protection system of wind turbine towers

SUMMARY

The coating systems used on offshore wind power production facilities are broadly acknowledged to be too good in the sense that the corrosion protection system is still in very good condition at the time of decommissioning the facility. It seems thus possible to reduce the cost of coating application by reducing the demands to the coating system and coating application. An industry group has been set to formulate a new standard for corrosion protection of wind power installations and the current work is part of the documentation package for the standard.

The members of the group designing the new standard are: Dansk Standard, FORCE Technology, Siemens Gamesa Renewable Energy A/S, Titan Wind Energy (Europe) A/S, Vattenfall, Vestas Wind Systems A/S, Welcon A/S, Hempel A/S, Teknos A/S, Cotes A/S and Wind Denmark.

The testing plan accounted for in this document is further funded by Siemens Gamesa Renewable Energy A/S and Wind Danmark.

OBJECTIVE / PURPOSE OF PROJECT

The aim of this project is to align the Danish wind industry on a cost-efficient corrosion protection system for towers in offshore wind energy. The work is part of an ongoing effort to standardize the surface protection systems used in the European wind sector.

The fundamental basis for the project is the notion that the currently used systems for towers are extremely good in regard to corrosion protection. Inspection of decommissioned towers reveals very few surface corrosion related issues and thus it should be possible to reduce the demands to the corrosion protection systems and thus reduce the cost of surface treatment.

It has been the ongoing notion that for enough long-term protection of offshore structures with duplex thermal spray zinc/aluminum (TSZA)/coating systems that a total film thickness of 300-500 microns is necessary. However, real-life experience on duplex systems has revealed very good performance on areas even with very low film thickness. Specific examples can be found on Tunø Knob Offshore Wind Farm where 180µm total DFT was measured and on now decommissioned and Vindeby Offshore Wind Farm where pinholes down to the TSZA layer were found with no apparent corrosion seen after 27 years of offshore environment. Also, the structural parts of Reefer Containers are coated with Duplex systems in very low film thickness with rather good result with regard to corrosion protection even though they are handled roughly with appreciable amounts of coating damage as result.

RESULTS & DISCUSSIONS

The aim to reduce the cost of the surface preparation and surface coating process by reducing the overall material consumption and time both in the surface preparation and coating process. Currently the demands for surface roughness, cleanliness and number of coats for offshore wind is more demanding than for other similar offshore structures and it is believed that the process is over-specified, and the lifetime achieved is much longer than necessary.

The initial part of this project is a thorough laboratory evaluation of a cutout of a decommissioned tower from the Vindeby Offshore Wind Farm as it provides a unique opportunity to investigate a ~27 year offshore exposed Duplex coating sample using advanced laboratory techniques. The main conclusion from this study is that very little corrosion is seen even on areas with pinholes and other defects. However, the UV protective topcoat is heavily abraded, chalked and discolored but still provides UV protection of the UV sensitive primer layers of the coating system. The complete tower after decommissioning is seen in figure 1.

Figure 1: Decommissioned tower from Vindeby Offshore Wind Farm Photo: Anders Nyboe

The current project aims at reducing the film thickness of metallization and coating, roughness of the steel and demanded cleanliness. Several new coating processes and specifications have been developed with a reduction in overall cost of 20-40% compared to the currently used system. In addition to the cost reduction a standardization of coating scheme would reduce the material loss at the tower manufacturers and at the same procedures can be followed even if the customer changes.

It is generally acknowledged that a film thickness of 280 microns is adequate for CX environments (offshore) according to ISO 12944-9 using zinc rich primers or thermal spray zinc coatings. This is done using Sa2½ cleanliness on medium profile sandblasted steel. The current tower spec calls for SA3 cleanliness and high blasting profile and a total film thickness of 380 microns. Years of experience has shown that the system performs exceedingly well beyond the necessary lifetime.

Furthermore, the increased use of environmental control inside the tower and nacelle reduces the need for corrosion protection on the inside surface of the towers during service.

The variables used in the test plan for the outside corrosion protection of the towers are the surface condition (roughness, cleanliness) before coating application and the film thickness of the corrosion protection system, both with regard to the thermal spray metallization layer and the epoxy and polyurethane coating layer on top. The surface protection systems have been tested according to a harsh cyclic test described in ISO12944-9:2018. This test has been the standard reference test for offshore oil and gas installations in Europe for the last decade.

Very similar systems have passed the test and are widely used on bridges and other offshore and coastal installations and thus the test was meant to only being able to distinguish between e.g. two different roughness profiles of the steel.

Half through the testing sequence the panels were thoroughly inspected revealing a rather unexpected failure mode resulting in corrosion-based delamination between the TZSA layer and the coating system. This was highly unexpected and regrettably prevented the differentiation between the tested surface protection systems. A representative panel is seen in figure 2.

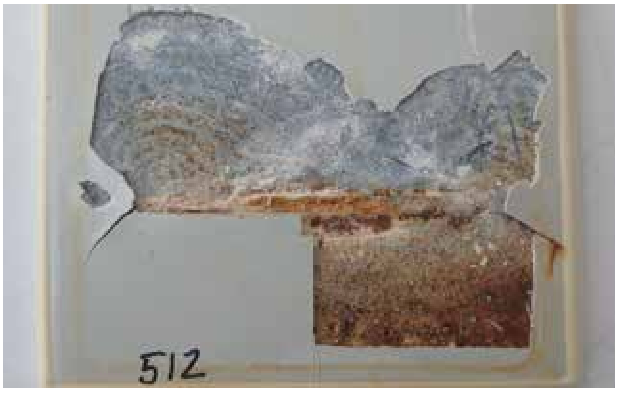

Figure 2: Heavy degradation of a Duplex coating system after ISO12944-9 cyclic ageing test

Final elucidation on why this failure mode is occurring is still ongoing on resources inside the Fast Track partnership but with funding from elsewhere. A premature, but highly likely mechanism of the failure is that the delamination is caused by galvanic corrosion resulting in anodic dissolution of zinc from the surface of the TSZA layer while the artificial damage to the steel substrate applied to the panel before exposure acts as a very effective cathode. The reason for this mechanism is with high certainty not found in the surface protection system as such but in the specific way the test was carried out. The most likely root cause is the specific way the artificial damage is created according to the relatively newly issued ISO12944-9:2018. The standard specifies a milling machined scribe through the organic coating and the metallization layer down to the steel substrate.

Earlier issues of ISO12944 allowed the use of a hardened steel scribing tool. This tool does not remove the complete layer of TLZA whereas a milling cutter affords clean steel. This results in rapid galvanic corrosion during the cyclic test due to the presence of the very active iron cathode surface provided by the milling cutter.

Similar tests run previously using the scribing tool has no active cathode surface due to TSZA residue and does no exhibit the corrosive delamination of the coating from TSZA. Other tests, including immersion in distilled water excludes defects from coating application such as dry spray, porosity and exceeded overcoating interval.

The above discussed phenomenon is still under investigation among the Fast Track partners under strict comparable conditions and while this work is not concluded yet, preliminary results verify the above conclusion. It cannot, however, be excluded that other factors such as the very low film thickness of the metallization layer could play a role in the spurious result seen.

The cost reduced systems for the internal surface of the towers are tested according to C2 or C3 corrosion category depending on the use of dehumidifiers in the towers. These coatings seem to pass the test requirements for the required corrosion and durability category. The tested surface protection solutions are 1- or 2-layer epoxy coatings without the use of TSZA and does thus not suffer from the same galvanic corrosion mechanism as seen above.

CONCLUSION

It is beyond doubt that corrosion protection based on Duplex systems using TSZA together with epoxy and polyurethane based coatings has shown extremely good performance in real life applications even though the film thickness is below specifications. However, the cyclic accelerated test used in the current project, degrades the Duplex corrosion protection system very rapidly. Thus, one take away message from this project is always to be cautious about correlating highly accelerated artificial exposure testing with real life results.

The Vindeby towers were in excellent condition at the time of decommissioning with very few coating damages and very few active corrosion spots. However, the topcoat was heavily degraded. Thus, from a performance point of view the conclusion is that for further extended lifetime a better topcoat such as a high-quality PU or polysiloxane should have been used. A refurbishment of the towers – if not decommissioned – could have been accomplished by high pressure washing and recoating with polyurethane topcoat.

The wind energy sector is under massive pressure regarding the total cost, from production of wind turbines to cost of ownership. The current project has been a part of the journey to reduce the cost of tower section production by not over-specifying the surface protection system.

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Duration of project:
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Collaborating companies:
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Fast Track 12: SolarSack “solar water disinfection”

SUMMARY

The aim of the project is to understand the degradation mechanism in different polymer foil, used for water purification technique process. The overall problem with the foil is, that it shall transmit the UV-A and UV-B rays through the foil and not degrade the foil.
In this project different types of foil are tested with and without water in the bag, up to 600 hours in a weather o meter.

OBJECTIVE / PURPOSE OF PROJECT

The purpose of the project is to find and test material combination of the transparent foil, that fulfills the criteria below:

- Increased SODIS (Solar Water Disinfection) efficiency (through improved UV transmittance)
- Increased durability (through improved resistance to UV degradation/weathering) equaling ~6 months product lifetime.

RESULTS & DISCUSSIONS

The reason for this test series is, that it is not possible to buy foil with lifetime data, for the operating conditions of this product.

UV Degradation/ Weathering

The samples are exposed to fluorescent UV lamps under controlled environmental conditions (temperature, humidity and/or water). Factors considered for experimental setup are:

a) type of fluorescent UV lamp; b) irradiance level; c) temperature during the UV exposure; d) type of wetting; e) wetting temperature and cycle; f) timing of the UV/dark cycle.

Natural sunlight near the equator (i.e. Uganda) will be recreated as closely as possible, with temperature and humidity/ wetting control to be discussed further.

We aim for SolarSack to be able to be used for 150 cycles of 4 hours (or around 5 months), in this sense we would ideally test this using an equivalent accelerated testing set up. A control polymer with well documented degradation performance could also be included for comparison.

The result of the UV Degradation / Weathering test is not as good as expected.

The first result from the UV degradation of the foil is, the foil turns golden relatively quickly. Hence, the time it takes to clean the water to be able to drink it is considerably extended.

The next result shows significant difference in the UV degradation speed, if the plastic foil is in contact with water on the inside or not. So, the temperature is an

important factor in this case.
In the picture to the right is the result of two different foils before and after 600 hours UV degradation.

Degradation of polymers

- UV
- Heat
- Moisture

Accelerated weathering

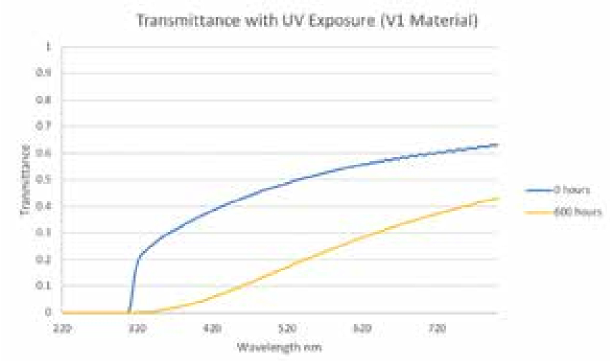
- Water filled samples

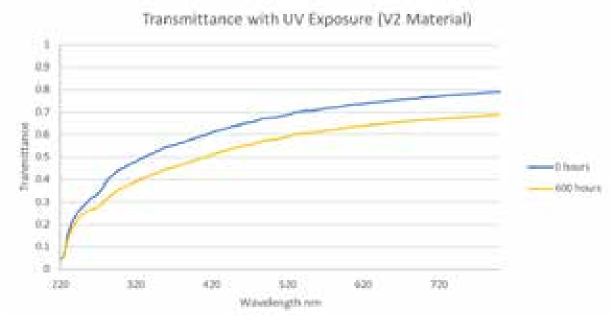
Factors of durability

- Reduction in UV transmittance
- Change in mechanical properties.

In the two diagrams is it possible to see that material V1 is more degraded, and the result is the UV transmittance after 600 hours is much lower than V2.

The mechanical properties after the UV degradation is in many cases already cracked. There must be made mechanical test of a few types, and this result is being used to make a decision for the next test program.





CONCLUSION

The conclusion after the first test series is, that it is more difficult than expected to have a life time of 6 months as well as an acceptable UV transmittance.

Therefore, a new test series with a new foil product has been started after this project. The new test series hopefully result in a product that fulfills the specification.

VALUE / IMPACT

The value for Solarsack is as follows:

Solarsack cannot produce a product that is not safe to use risking bad publicity and complaints.

After more test series Solarsack can produce a product that has a documented lifetime and better performance.

For products of this type, intended for sale to relief organizations, it is important to ensure that the product meets the specification, and is safe to use - that is, if the instruction is followed, the water is clean after the specified time.

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Duration of project:
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Collaborating companies:
SolarSack, FORCE Technology

Fast Track 13: TRD wear testing

SUMMARY

A test setup and method have been devised for abrasive wear testing of spray nozzles. In partnership with the sme TRD Surfaces, comparative wear testing of nozzles with and without a special gas phase coating from TRD has been performed, to assess and demonstrate the wear characteristic of the TRD surface treatment on low pressure flat spray nozzles. Through this specialized test is has been demonstrated that the TRD surface treatment significantly improves the wear rate of nozzles.

OBJECTIVE / PURPOSE OF PROJECT

The purpose of the project is to:

- Modify existing high-pressure test rig to fit with 10 bar nozzles, and demonstrate consistency of test
- Perform parameter study by iterative test runs and evaluation of wear rate
- Perform comparative wear test of flat spray nozzles with and without wear reducing surface treatment
- Evaluation of nozzle wear by scanning electron microscopy.

RESULTS & DISCUSSION

The research spin-out startup company TRD Surfaces offer a gas-based surface treatment of metals. One suitable application of the gas treatment is to improve the wear resistance of flat spray nozzles in the range of 10-100 bar pressure. The treatments have shown to increase the surface hardness, but actual application testing and characterization, which end customers require, are beyond the capabilities of TRD Surfaces.

If the gas treatment can reduce nozzle wear for common 10 bar nozzles, and thereby reduce water and chemical consumption, there is a clear business case for several industries for upgrading cheap nozzles with the gas treatment.

FORCE Technology is in the possession of a wear test rig for flow tests and this has on an experimental basis been used for wear test of spray nozzles at high pressure (100 bar).

In the project the test rig has been modified and fine-tuned for wear testing at 10 bar of flat spray nozzles with a new surface treatment and it has been demonstrated to be consistent and effective method for testing abrasive wear of nozzles.

The test rig consists of a high head pump, circulation spray chamber and a volumetric measurement device, capable of dividing and measuring the volumetric flow rate of water across the spray fan, see figure 1.

By adding diatomaceous clay at various concentrations to the circulating water in the test rig, accelerated wear of nozzles and valve surfaces can be achieved, where wear will increase as a function of increased clay-concentration in the solution.

Using a clay-concentration of 6 g/l comparative wear tests of flat spray nozzles with and without wear reducing surface treatment from TRD Surfaces could be performed within a reasonable timeframe, showing a significant effect of the surface coating on the wear-rate of the nozzles, see figure 2.

Further evaluation and analysis of the wear surfaces of the tested nozzles using scanning electron microscopy is to be performed by DTU.

Figure 1 Left: Spray chamber, with 3 flat spray nozzles during a test. Right: Close up of a untreated flat spray nozzle

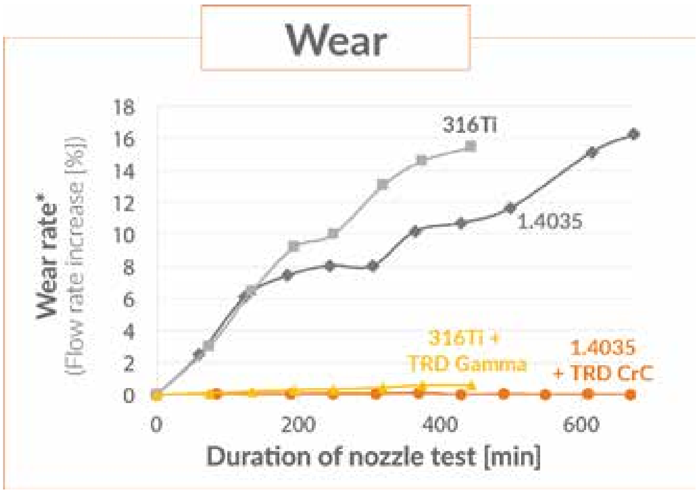

Figure 2: Accelerated wear tests of nozzles. Diatomaceous clay was added to speed up wear.
**316Ti tests: 10bar, 6 g/L abrasives - 1.4035 test: 100bar, 2 g/L abrasives*

CONCLUSION

A test setup and method for low pressure abrasive testing of spray nozzles have been devised. It has been demonstrated that the test setup and method can produce consistent measurements. By comparative testing of untreated reference flat spray nozzles and nozzles with a TRD surface coating, it has been shown that the TRD surface coating resists wear as opposed to the untreated nozzles that, as result of wear during testing experienced a 15 % increase in flowrate.

VALUE / IMPACT

It has been shown that the TRD surface treatment can reduce nozzle wear for common 10 bar nozzles, and thereby reduce water and chemical consumption. Hence, there is a clear business case for several industries for upgrading cheap nozzles with the gas treatment, and thus reduce water consumption and energy consumption for pumping.

The test rig has been demonstrated to be consistent and effective at nozzle testing at both 100 and 10 bar, and furthermore, the test rig has also shown to be effective in testing wear of valves with variable opening. The test rig will be relevant for agricultural, pharmaceutical, oil & gas and a range of other industries, in which material optimization could save resources – energy, water and material consumption. The direct impact for FORCE Technology is an improved response time in order to offer realistic tests, the long-term effect is opening new markets for material producers.

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Duration of project:
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Collaborating companies:
TRD Surfaces (TRD), Technical University of Denmark (DTU), FORCE Technology

Fast Track 14: DANCOP - improving safety impact barrier

SUMMARY

The project aims at delivering materials consultancy and testing to the project owner (DANCOP) towards improving one of their products. Specifically, they wished us to improve key properties of a safety impact barrier: Making it stronger, more flexible and able to retain its shape at elevated temperatures, e.g. when used in warmer climates. All improvements may only impose small cost increases and should not constrain the ease of product assembly.

OBJECTIVE / PURPOSE OF PROJECT

The overall purpose of the project is to improve the DANCOP A/S safety barrier in the following areas:

- Increased impact resilience
  - Increased strength
  - Increased flexibility
- Increase shape stability towards higher temperatures and direct sunlight.

RESULTS & DISCUSSIONS

The aim with the project is to make the material in the DANCOP barrier series more flexible in order to absorb more energy under impact. The material has to retain memory which allows the material to go back to its original form after impact.
DANCOP has created a new product series of flexible impact barriers. To improve the product further, they wish to optimize energy absorption upon impact and increase the overall impact resilience. For this they have been looking at different parameters to change and how to implement smart solutions. This is where FORCE Technology comes in.

This project has consisted of a combination of methods:

- Materials consultancy: In written and verbal form, on the basis of what was explained by DANCOP, and what is the known "best practice" by FORCE Technology´s specialists.
- Computer aided mechanical simulations: In order to establish a firm base of knowledge on the mechanism of energy absorption of the existing barrier design. And from which to test and recommend changes to the design.
- Prototype design and testing (ongoing).

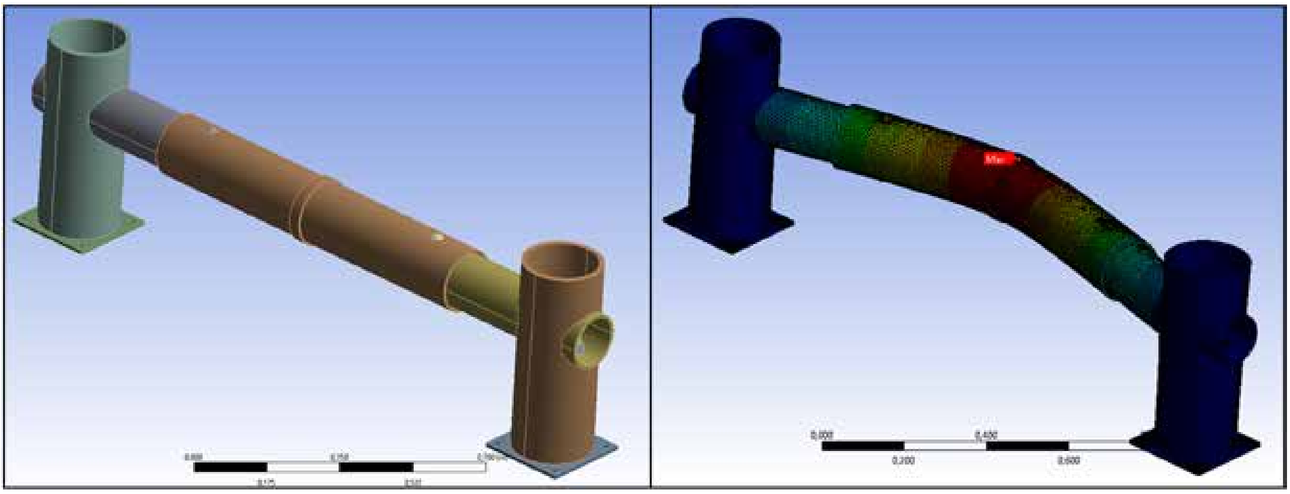

Figure1: The safety barrier. Left shows the computer model of the structure before deformation, while the image to the right shows the barrier after the simulated impact.

The results of the computer simulations (example seen in figure 1) gave valuable insights on the mechanism of energy absorption. Most importantly, it underlined the important balance of material flexibility and strength. The simulations thus enabled the specification of a set of design criteria, from which to develop product improvements.

On the basis of the above described insights, FORCE Technology has outlined a set of design recommendations which serve the dual purpose of absorbing impact energy while also stabilizing the structure: Both in terms of shape retention after impact and also stability towards exposure to higher ambient temperatures and direct sunlight.

CONCLUSION

On the basis of the computer aided mechanical simulations of the safety barrier, a set of design recommendations were given, that serve to augment the strong suits of the existing product by retaining the right balance between strength and flexibility. These recommendations will hopefully lead to better impact resilience of the safety barrier, while also improving the product in terms of shape retention and structural stability towards higher temperatures and direct sunlight.

VALUE / IMPACT

The recommendations to the improved safety barrier that have resulted from this project, increase the competitiveness of the product as it increases the impact resilience, and the advertised maximum energy that can be absorbed. Specifically, also the increased structural stability towards high ambient temperatures and direct sunlight, increases the appropriateness of the product towards large markets in the tropics and subtropics.

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Duration of project:
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Collaborating companies:
DANCOP A/S, Aalborg University (AAU) and FORCE Technology

Fast Track 15: Viable substitute to Chrome-VI surfaces in food zones and optimizing of surface tests

SUMMARY

The purpose of the project is to identify a viable surface treatment for aluminum parts in food contact areas or to identify alternative solutions for the parts. Mechanical properties, cost and visual appearance are important decision parameters. The requirements for the parts and surfaces and the possible solutions were set in the Fast Track team. Based on the knowledge gained, surfaces were selected and parts for testing were made in collaboration with the partners, and the possibility of manufacturing the part in new materials was investigated. The surfaces were tested and evaluated according to the new surface and material test plan made in collaboration with the Fast Track partners. The results were very diverse but pointed towards a coating solution that could be applied on the aluminum parts.

OBJECTIVE / PURPOSE OF PROJECT

- Idea generation of possible surface treatments and solutions that can replace an existing Chrome (VI) surface used on parts in food contact zones.
- Get new surface treated test samples through involved partners.
- Evaluate the surface treatment properties by setting up appropriate test procedures.
- Optimizing and defining a test plan for selecting and testing of surfaces.

RESULTS & DISCUSSIONS

The project concerns a part in a food contact zone on a planetary mixer. It is the main part of the planetary gear and is visible from the machines outside – it is called the lower head.

The main aim of the project is to determine a surface treatment that can replace an existing Chrome (VI)-surface used on aluminum parts in food contact zones. The new surface should have a good visual appearance and reduce or in best case eliminate the risk of fractures of the surface treatment peeling off and into the food. A secondary aim was to optimize an existing test plan that should be defined for application in other development projects in the future.

The part was previously manufactured in China, but the production was moved back to Denmark, mainly due to quality issues. In this process we were set with a new challenge that Chrome (VI) is no longer an opportunity in Europe. The lack of access to Chrome (VI) initiated this project.

This led to the ambition that all Chrome (VI) surfaces should be replaced over time and the alternative should be applied on other products and in new development projects hence another drive for Varimixer to find a viable substitute.

Fast Track continues already initiated surface treatment test, the methodology building on scenarios imitating real conditions of use, e.g. in kitchens and bakeries.

The following test parameters were set in collaboration with the Fast Track partners, and by evaluating criteria as risk of impact when handling the tools used in the machine, cleaning, commercial appearance "how does it look?", etc.

The total manufacturing cost is another important parameter in order to be competitive and have a viable business case.

Test protocol:

- Washing test for 10 hours in an industrial dishwasher, with a "worst case" scenario, using high alkaline detergents (pH 8-11) at 60 C°.
- Wear test of the surface, impact test using equipment at FORCE Technology
- Setting up a new test plan based on all the criteria.

The surfaces and other materials that were selected and further investigated were listed at a kickoff meeting with the Fast Track partners. Following candidates were selected for further investigation, currently the surfaces need to pass the final impact test at FORCE Technology:

- Anodized, the surface treatment did not pass the washing test, pitting started to occur after 2 hours.
- Keronite 30µm, a ceramic surface that passed the washing test, but the visual appearance was not satisfying, and the surface structure gave cleaning issues.
- Teflon (PTFE) 60µm, passed the washing and wear test, the ductile surface showed good impact resistance, and it looks great. The downside of Teflon is the cost per part, with 43% being the coating.
- Powder-coated polymer surface. Tests are still being conducted, but show promising results so far, many similar properties as Teflon, but at a competitive price.
- CrN, very strong surface, passed the washing test with no defects. The surface is smooth, but unfortunately it was too expensive to be competitive in this application.
- NiSn 5µm, the surface needs further testing of its properties.
- Stainless steel, it was investigated if this was a viable solution, but due to process and manufacturing parameters, this solution is too expensive.
- Fiber-filled plastic; after sparring with SP Moulding A/S, it was concluded not to be a solution, due to the gear geometry affecting the strength combined with a low yearly volume of 1500 pcs.

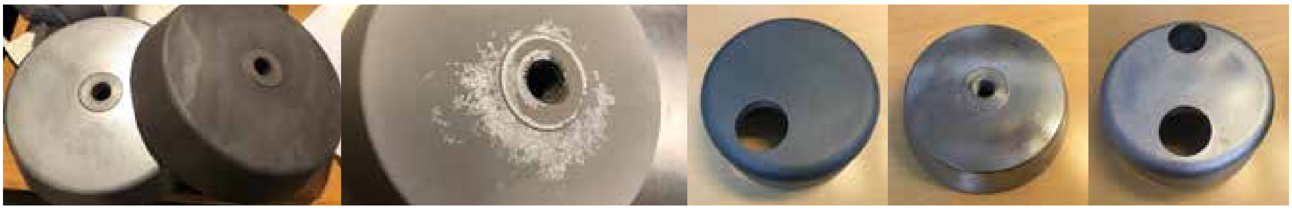

Figure 1: Surfaces showed in chronic order.

Figure 2: Accelerated wear test of the surface in a lathe.

CONCLUSION

The results from this Fast Track shows that the high-alkaline detergent is very aggressive even to surface treatments known as durable, resulting in corrosion and pitting at an early stage. It can be concluded that the surfaces with a galvanic coupling is not preferred in a setting like this – this was also stated by the Fast Track partners before initiating the project. The Teflon solution being thoroughly tested showed good crack resistance at impacts due to high elasticity, whereas the powder coating needs further impact testing before making any technical conclusions, but both surfaces are promising candidates.

The overall conclusion is that the two most important parameters are resistance to detergents and impact strength, and that it is possible to find other technical surfaces that can replace chrome (VI).

VALUE / IMPACT

We have reduced the most important risk which is the use of the hazardous chrome (VI) as well as the risk of the material peeling off.

From the Fast Track results and knowledge gained we expect to reduce the total cost per finished unit to less than the manufacturing cost in China. This could increase our margin on three machine series.

The results from developing a guide for selecting and testing new surfaces and materials in food contact zones, are reflected in new guidelines that are being incorporated in our development process.

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Duration of project:
September – December 2019

Collaborating companies:
DANCOP A/S, Aalborg University (AAU) and FORCE Technology

External suppliers:
Dancolor, Accoat

Photo courtesy of LM Windpower

Fast Track 16: Degradation of GFRC materials from wind turbine blades prepared from polyester and epoxy-based binder systems through subcritical water based solvolysis and supercritical carbon dioxide

SUMMARY

Epoxy and polyester based composites are becoming more and more abundant in various consumer product from wind turbine blades, boats, cars and various appliances. This is generating an increasing amount of waste on both short and longtime, almost with an exponential growth rate. This creates an eminent demand for decommissioning, waste handling and possible recycling solutions of thermoset binders that are very difficult to degrade and recycle.

The project is an extension of the previous GENVIND where various recycling paths were tested.

The current project has tested polyester based GFRC supplied by LM and epoxy based GFRC supplied by Siemens Gamesa Renewable Energy A/S.

The samples were exposed to subcritical water solvolysis at FORCE Technology and supercritical carbon dioxide solvolysis at Danish Technological Institute.

The experiments verified that it was possible to decompose both polyester and epoxy-based binder systems in subcritical water based solvolysis. The sub critical water solvolysis also verified that the chemistry of the changes with increasing temperature, pressure and duration of the process.

It was possible to decompose polyester based binders in super critical carbon dioxide using combinations of co solvents to the process.

OBJECTIVE / PURPOSE OF PROJECT

- Testing the possibility to decompose polyester and epoxy-based binder systems using both supercritical carbon dioxide solvolysis and sub- to super critical water solvolysis
- Testing the possibilities to combine subcritical digestions of the above binder systems with super critical carbon dioxide digestion.

RESULTS & DISCUSSION

Introduction:

Polymer based industrial and consumer products are becoming more and more abundant and the problems related to proliferation, recycling and degrading is resulting in increasingly public awareness.

Many thermoplastic polymers like polyethylene and polypropylene products can more or less be recycled directly whereas the thermoset plastics like polyester and epoxy will have to be incinerated in order to break down the structure. Thermoset polymers can be decomposed using either pyrolysis or various forms of solvolysis using either sub- to super critical water solvolysis or solvolysis using carbon dioxide, co solvents or solvents like various alcohols, acids, alkalines and catalysts. The efficiency of the process is reliant on the temperature/pressure/duration profile which will also govern the possibility for synthesis during the process. The possibilities for controlling the degradation and possible subsequent synthesis of the derivate might be adventitious in reusing the materials.

Water based solvolysis

The experiment was performed using a PARR 4653 reactor with a reactor volume of 1000 ml. The reactor can operate at super critical conditions 374°C/220 bar. It is possible to take samples of the fluid during the operation. The temperature and pressure can be monitored and adjusted on the control board using PID controllers.

Samples of polyester and epoxy-based glass fiber were sectioned into 140 mm x (17-37 mm) x 11 mm sticks that could be fitted into the reactor.

Based on previous experiments during the GENVIND project it was estimated that the polyester based binders in the LM samples would start to degrade at around 275°C and the material would be completely disintegrating at around 300°C. The epoxy-based binders were expected to start degenerating around 300°C and to be completely disintegrated around 350°C.

Test 1

Test 1 was conducted on samples from LM. 206 grams of polyester based glass fiber and 400 gr. of demineralized water were placed in the reactor, and the reactor volume was purged with nitrogen for approximately 10 minutes. The reactor was ramped up to 274° C in 45 minutes and a pressure of 62 bar. At 228°C a weak scent of styrene could be detected, and the scent increased until final temperature at 274°C where the heat was shut off and the reactor was left until the following morning.

The reactor was drained the next morning and the fluids were collected. Fluid was clear and reeked of styrene. The fiberglass fragments were still coherent, but porous like wood.

Test 2

Test 2 was conducted on samples from LM. 197,5 grams of polyester based glass fiber and 401 gr. of demineralized water was placed in the reactor, and the reactor volume was purged with nitrogen for approximately 10 minutes. The reactor was ramped up to 304° C in 73 minutes and a pressure of 95 bar. The temperature was held for 25 minutes and subsequently the heat was turned off.

The reactor was drained the next morning, and the fluids were collected. Fluid was clear and had an estery scent. The fiberglass fragments were still coherent, but porous, expanded and cracked like wood.

Test 3

Test 3 was conducted on samples from Siemens. 197 grams of epoxy-based fiberglass and 400 gr. of demineralized water was placed in the reactor, and the reactor volume was purged with nitrogen for approximately 10 minutes. The reactor was ramped up to 325° C in 110 minutes and a pressure of 120 bar. The temperature was held for 25 minutes and subsequently the heat was turned off. A reactor fluid sample was taken after 120 minutes after heating off. The fluid was multiphase with both an oil phase, water phase and sticky precipitated phase.

The reactor was drained, and the fluids were collected. The reactor fluid was multi phased as observed in the in-process sample. Fluid had a pungent amine stench and it was slightly sticky. The fiberglass fragments were completely dissolved, and the individual fibers could be peeled out. The fibers could be completely cleaned by acetone leaving them free and bright. The acetone extracts were dark brown and sticky, and it was collected for analysis together with the water phase.

Test 4

Test 4 was conducted on samples from Siemens with the purpose of just hitting the P/T value where the epoxy would start to degenerate. 202 grams of epoxy-based fiberglass and 401 gr. of demineralized water was placed in the reactor, and the reactor volume was purged with nitrogen for approximately 10 minutes. The reactor was ramped up to 300° C in 120 minutes and a pressure of 85 bar. The temperature was held for 25 minutes and subsequently the heat was turned off.

The reactor was drained, and the fluids were collected. The reactor fluid was again multi phased as observed in test 3. Fluid had a pungent amine stench and it was slightly sticky. The fiberglass fragments were completely dissolved, and the individual fibers could be peeled out. The fibers could be completely cleaned by acetone leaving them free and bright. The acetone extracts were again dark brown and sticky, and it was collected for analysis together with the water phase.

CO_2 based solvolysis of cross-linked polyester based wing material

Initially the glass transition temperature and the polyester were determined by TGA. Tg was found to be 115 degree Celsius. The experiments carried out in supercritical CO_2 reactor is summarized in Table 1.

Table 1: Experiments on polyester based wing material carried out in supercritical CO_2 reactor

Material	Reactant (g)	Temperature (˚C)	Time (h)	Weight loss (%)	Comment
Polyester	No	100	24	<1	No reaction
Polyester	1:2	100	24	<1	No reaction
Polyester	1:2	130	24	<1	Surface reaction (Fig)
Polyester	1:10	130	24	35	3D reaction (Fig)
Polyester, pretreated in hydrothermal reactor	No	100	4	2.5	Styrene extracted during treatment

Fluid Analysis

The fluids extracted from the experiments were analyzed using SPME technique to screen the composition.
The screening shows that the composition of the reactor fluids is very complex, reflecting combinations of various breakings of the polymer structure into raw monomers and synthesis of the decomposed molecules.
Since the whole decomposition spectrum has not been tested it is difficult to predict the degradation path of the derivates with increasing P/T ratios.

CONCLUSION

Based on the four reactor experiments at subcritical water solvolysis on polyester and epoxy-based fiber-glass samples and subsequent SPME screening of reactor fluids the following can be concluded:

- The previously reported findings about degradations of epoxy and polyester based polymers were confirmed in this study. The polyester based polymers start to decompose around 220°C and accelerates around 300°C with subsequent initial synthesis of monomers. In this study it was found that the epoxy used in the SIEMENS fiber-glass composite start to decompose around 275° and the decomposition almost complete around 300°C leaving the fibers free and easily cleanable in acetone. The previous experiments with epoxy in the GENVIND project showed that the decomposition usually started around 300°C. The differences in the degradation temperature is most likely related to the initial formulation of the epoxy in the various experiments.

- Polyester based wing material can be decomposed by solvolysis in a supercritical reactor as illustrated in Figure 3. The Process can be commercialized by maturing the solvolysis chemistry and upscaling the reactor technology.

VALUE / IMPACT

The experiments show that that it is possible to chemically degrade thermoset polymers using both individual solvolysis using water and super critical carbon dioxide or combinations of both processes at temperature and pressure conditions that are commonly used in the industry. This opens an opportunity to perform advanced degradation of polymer waste, where the biproducts are either further refined or used directly as fuel or raw materials for other chemical processes. It is possible that by combining different degradation processes that these methods can be refined to a level where the P/T conditions can be further reduced and that it will be possible top target specific compounds by optimizing the synthesis reactions during the process.

The understanding of how polymer materials behave under extreme P/T conditions offers an opportunity to test and optimize sealing compounds and coatings to specific aggressive environments where various fluids and combinations of fluids operate either under static or dynamic conditions.

Figure 1 PARR reactor setup

Figure 2 Raw polyester based fiberglass sticks from LM

Figure 3 Example of decomposed polyester-based fiberglass after sub critical water solvolysis from LM

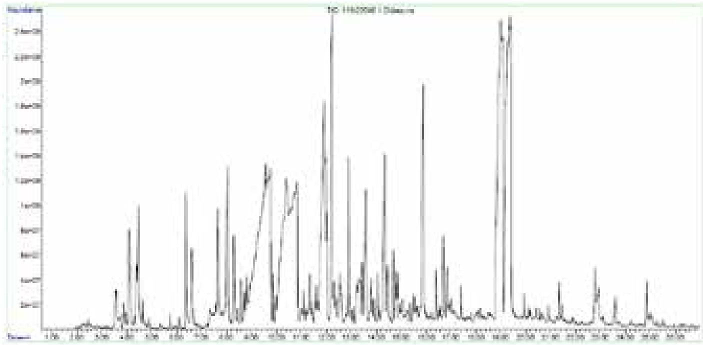
Figure 4 Reactor fluids from test 1 and 2

Figure 5 Fiber extracted from reactor before and after cleaning

Figure 6 Reactor fluids from test 3 and 4

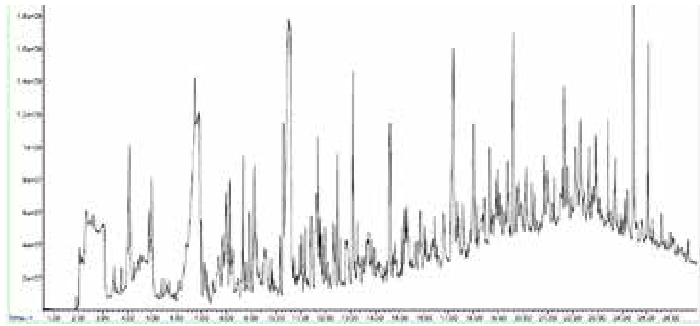
Figure 7 Polyester based wing material loaded in super critical CO2 reactor

Figure 8 Wing material after treatment in supercritical reactor with a reactant ratio of 1:2 at 130 C. Surface reactions has occurred.



Graph 1: The chromatogram from the GC-MS shows the peaks detected in the water phase using the SPME technique to extract organic phases in the reactor fluids from the experiment on the polyester based fiberglass in experiment-1.

Figure 9 Wing material after treatment in supercritical reactor with a reactant ratio of 1:10 at 130 C. 3D decomposition has occurred.



Graph 2: The chromatogram from the GC-MS shows the peaks detected in acetone dissolved heavy brown phase using the SPME technique to extract organic phases from the experiment on the epoxy-based fiberglass in experiment-3. The chromatogram indicates a very wide array of primary and secondary polymers, including complex amines

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Duration of project:
October 2019 – December 2019

Collaborating companies:
Siemens Gamesa Renewable Energy A/S and LM Wind Power

Fast Track 17: Optimising material selection in connection with biodiesel production

SUMMARY

The Fast Track project aimed to gain knowledge about the corrosion process in the pre-esterification process at Daka eco-Motion, who manufacture biodiesel from slaughterhouse waste. Literature screening and laboratory testing at process conditions confirmed that stainless steel 904L is the best suited material for reactor vessels in this process. Dry grinding of stainless steel reduces the corrosion resistance of the alloy, by what is presumed to be local heating of the alloy.

OBJECTIVE / PURPOSE OF PROJECT

- Gain an understanding of the corrosion process in this particular process environment
- Look for similar solutions in similar conditions in literature
- Suggest potential alternative materials, coatings, etc.
- Test existing and alternative solutions in a similar environment.

RESULTS & DISCUSSIONS

Daka ecoMotion produces biodiesel from animal fats derived from slaughterhouse remains. The raw product is a mixture of free fatty acids (FFA), triglycerides and some impurities.

The project concerns the primary process step in the biodiesel production where FFA's are esterificated using methanol and sulfuric acid at 75° C in a 75 m^3 stainless steel 904L vessel.

At present corrosion pits have formed primarily in the bottom of the vessel, with costly repairs consequently.

The projects main aim would be to simulate the process step in the laboratory and to test different stainless-steel alloys with regards to corrosion.

Additionally, suitable surface treatments e.g. thermally sprayed metallic coatings, electrolytically deposited coatings or inorganic coatings could be tested in real life conditions.

The end result would give Daka ecoMotion valuable knowledge on the corrosive properties of the esterification process step and provide guidance as to which materials and/or coatings are suitable for the production equipment.

We performed a literature screening combined with suggestions for alternative coatings and finally testing of different alloys in similar conditions to replicate the corrosive conditions in the vessel.

However, it was not possible to find the exact mixture of sulfuric acid, methanol and animal fat at 75° C in combination with stainless steel 904L in the literature search.

The closest match in materials section guides was 96% sulfuric acid at 80° C. At these conditions stainless steel 904L is the best choice as the lower alloyed variants such as 304 and 316 have higher corrosion rates. Higher alloyed stainless steel types such 6Mo have similar corrosion properties and do not warrant usage due to the higher price. Nickel based alloys, such as Inconel 625 perform similar to the lower alloyed stainless steel types and are also not recommendable.

One source, however, indicated that dry grinding has a negative effect on the corrosion resistance of stainless steel. This is consistent with some of the actual corrosion attacks observed in the vessel. The cause for this phenomenon is not known but it is assumed that the temperature reached during grinding has a negative effect on the corrosive properties of the alloys.

Alternative solutions, such a chemical deposition and thermal spraying of nobler metals were omitted due to concerns for porosities in the layers which would have a detrimental effect on the corrosive properties of the system with sulfuric acid at 75° C.

A sample of the most corrosive liquid from the vessel, the bottom fraction containing 96% sulfuric acid, methanol, and impurities, was used for testing with various alloys at the process temperature of 75° C.

Polarization curves for the different alloys were obtained and have provided the project with valuable information about the corrosion process with various alloys.

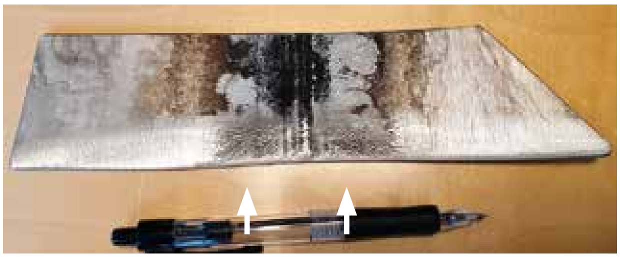

Figure 1: Sample from old vessel with pitting corrosion in the vicinity of a weld.

CONCLUSION

The project has yielded the following results:

- Valuable knowledge of the corrosion process in biodiesel production has been obtained
- Stainless steel 904L seems to be the best candidate from a corrosion and cost perspective
- Alternative coating techniques, such as electrochemical deposition and thermal spraying are not immediately suitable for this application.

VALUE / IMPACT

The biodiesel manufacturer has obtained valuable knowledge with regards to the corrosion process of the pre-esterification process at their plant. This can be used to minimise the risk of unscheduled production stops and there by production losses. Furthermore, maintenance can be optimised, expenses saved, without compromising asset integrity.

FORCE Technology has gained valuable information about the specific issue which can be utilized in similar applications, to the greater good of society.

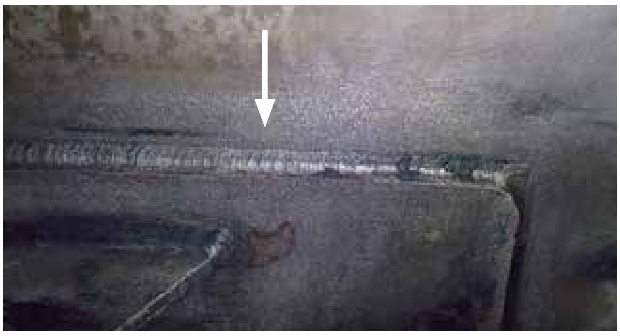

Figure 2: Image from the current vessel with a rough appearance in the vicinity of a weld. This could be an early indication of pitting corrosion.

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Duration of project:
October 2019 – December 2019

Collaborating companies:
Daka ecoMotion, FORCE Technology

Fast Track 19: Current collector for electrochemical cell

SUMMARY

HPNow is pioneering a novel technology for electrochemical hydrogen peroxide generation. The technology is based on proprietary catalyzed membranes that generate hydrogen peroxide with oxygen, water and electricity. To establish an electrical potential at the electrodes, a current collector is inserted in the electrochemical cell housing in direct contact with the electrodes. This current collector needs to have specific dimensions to meet with the mechanical design criteria, and it needs to withstand the harsh operating environment of the electrochemical cell. In this project, suitable manufacturing methods for this part were investigated in collaboration with DTI and FORCE Technology.

OBJECTIVE / PURPOSE OF PROJECT

To date, HPNow is using an expensive 3D printing technology for production of the current collectors, which presents limitations in terms of cost and scalability. In this project we are looking for an alternative production process in order to achieve:

1. A suitable production method to cost down the part.
2. A scalable production method with high capacity.

At the same time, the performance of the current collector should be long-term stable and show the same low voltage performance as the expensive 3D printing electrodes.

RESULTS & DISCUSSIONS

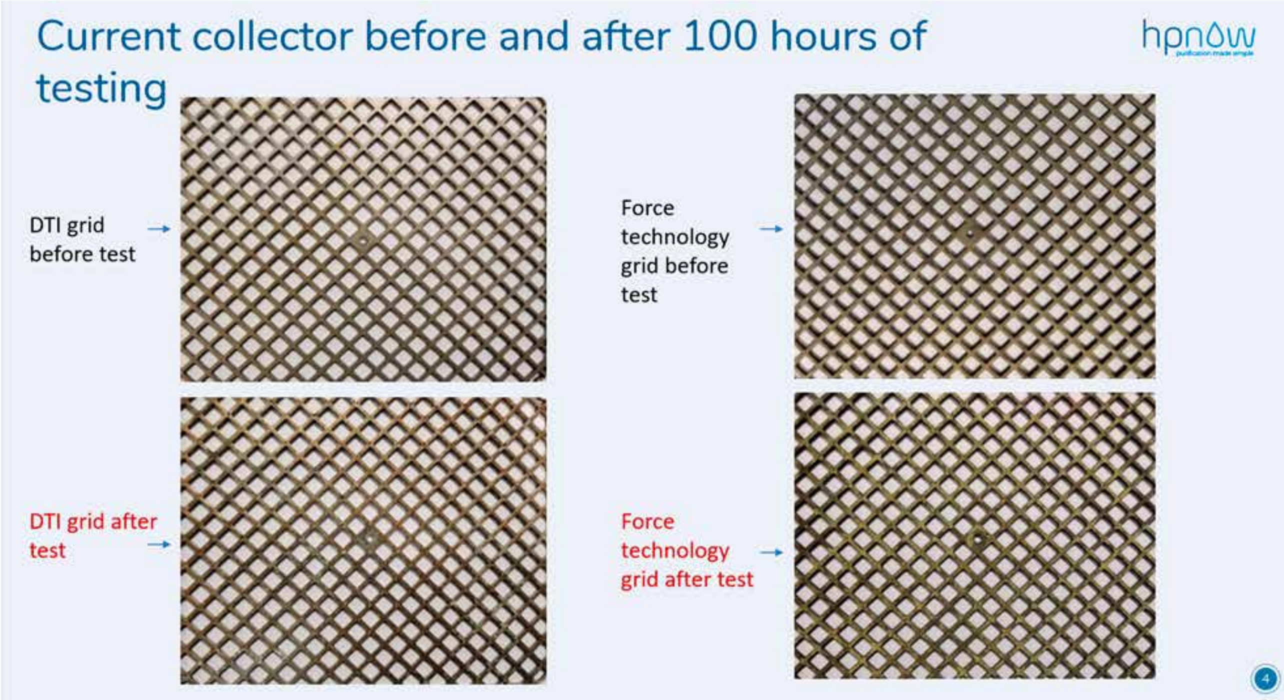
HPNow has tested variations of the current collector in terms of materials and production method. In particular, we have tried using aluminum as a base material (which had corrosion issues). At the onset of the project, the parties held a meeting to brainstorm possible solutions. The leading approach was to coat aluminum grids with titanium, given this would have the potential to meet both the cost as well as the scalability targets. Both DTI and FORCE Technology have inhouse capabilities to coat the aluminum base with titanium based on PVD technology and plasma spraying. The core idea was that titanium would form a protection layer and prevent the aluminum substrate from corroding. While the coating improved the situation, corrosion was still heavy, and the parts had a lifetime of a few tens of hours only (see picture 1). Analysis on the root cause for corrosion is underway and is expected to be complete within a few weeks.

CONCLUSION

The parts produced and tested have not been successful in our tests and cannot be implemented due to heavy corrosion of the parts. During the project, some additional ideas for other suitable manufacturing methods as well as alternative solutions came up that may be explored at a later stage.

VALUE / IMPACT

For PVD and plasma-sprayed Ti there will be no impact due to corrosion of the underlying Al substrate.



Picture 1: Current collector before and after 100 hours of testing

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Duration of project:
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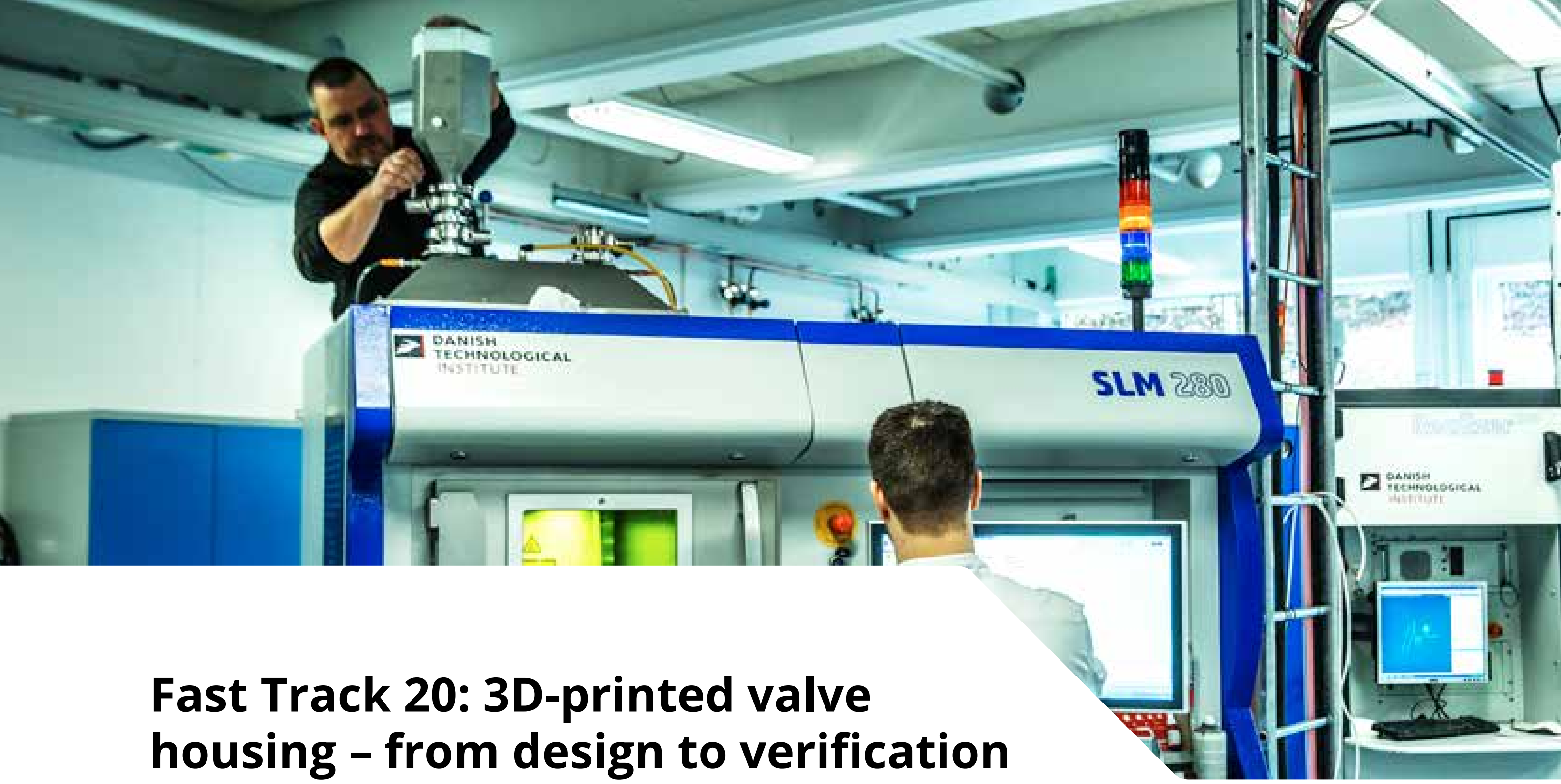
Collaborating companies:
Danish Technological Institute (DTI), FORCE Technology



Fast Track 20: 3D-printed valve housing – from design to verification testing

SUMMARY

The Fast Track identified the market potential in compact valve housings with less pressure losses due to flow restrictions. A new valve housing has been designed for metal additive manufacturing (AM/3D-print). It is, however, essential that the valve housing can withstand the required pressure and cyclic operation, hence this is also included in the Fast Track.

The valve housing was designed, built and tested according to the requirements of the existing conventionally manufactured valve housings.

OBJECTIVE / PURPOSE OF PROJECT

The aim of the FastTrack project was to clarify if metal AM is a viable solution for Danitech (DT) with their current business case, as well as future applications. With assistance of Danish Technology Institute (DTI) and FORCE Technology (FT).

3D-printing the valve housing for various hydraulic applications will allow DT to customize, reduce weight, save space, increase performance, and reduce lead times of the valve housings. This will give DT a competitive advantage in its local markets and open for new types of hydraulic products. Secondly, it will eliminate the need for expensive casting moulds (for high pressure die casting) and thereby giving DT valuable flexibility in the design and production phases.

The scope of the project is:

- Design a new valve housing (DT and DTI)
- Design a pressure test that best describes and analyses the required application's functionality (DT and FT)
- Manufacture the valves determined by the pressure test design (DTI)
- Test the valve housings according to the pressure test design and deliver performance documentation for the valves tested. (FT)

A project description has been created between the three parties, outlining the scope and activities. Based on the specific business case at hand, the functionality of the hydraulic valve housing was then presented for DTI and FT. Subsequently, the various design options were then discussed between DT and DTI. This led to, after two iterations, a usable demo-design of the hydraulic valve housing.

The goal of the design was to create something that was compact and easy to 3D-print. The design of the valve housing has been optimized for metal AM in AlSi10Mg and was modelled in SolidWorks and Magics.

RESULTS & DISCUSSIONS

Traditional casting dies have been tried and used. However, creating a new set of dies can be very expensive and the payback period is long due to the low quantity DT requires.

DT has participated in the AM Sustain program hosted by AM Hub and Deloitte and as part of the program identified a valve housing application that can make a good business case for their company. The available solution is cumbersome and expensive. A 3D-printed

valve housing can be more compact and potentially also cheaper. Documentation that the 3D-printed valve housing can withstand the required pressure is needed to proceed with the business case.
The valve housing was designed with the DfAM principles and according to the valve housing design rules used by DT for their conventional parts – meaning 7 mm wall thickness around all pressure carrying parts. Additionally, the part was constructed by defining the functional interfaces and using the minimal part design. See picture 1. It is believed that a more compact design can be achieved by overlapping the geometry of the pressure carrying walls, however, for the first iteration a conventional and safe design was selected. In many respects the main limiting factor regarding the compactness of the design is the valves themselves, and the room needed to thread the mount as well as ensure external connectivity for the valves.

The design was developed as a collaboration between DT and DTI. Following the design phase, four hydraulic valve housings were 3D-printed in Aarhus on DTI's SLM Solutions 280 printer. The material used was AlSi10Mg. The parts were not heat treated. It was not possible to procure the special valve milling tools within the short time frame of the Fast Track project. Hence, it was only possible to make an ultimate pressure capability test on the complete part.

Four parts were built and shipped to FT for subsequent pressure testing.

The pressure test performed at FT was performed as a manually operated test, i.e. it was not possible to carry out a high-cycle test to verify the fatigue performance of the design within the frames of this project. The test was thus initially performed with 25 cycles from 0 to 250 barg to verify durability at operational pressure. Subsequently, two ultimate pressure tests were performed. The first test to verify the system performance to four times the operational pressure, i.e. 1000 barg. This test was followed by a test-to-leak that showed durability up to 1,490 barg at which the threads in the aluminium started to creep leading to leaks from the plugs.

The project has shown that production of AM valve housings can be a way forward and that the robustness of printed specimens is promising. We expect that the part can be further optimized for AM.

CONCLUSION

The valve housing was successfully designed for metal AM and was built in four copies for subsequent testing. We expect that the part can be further optimized for AM following the end of the Fast Track project.

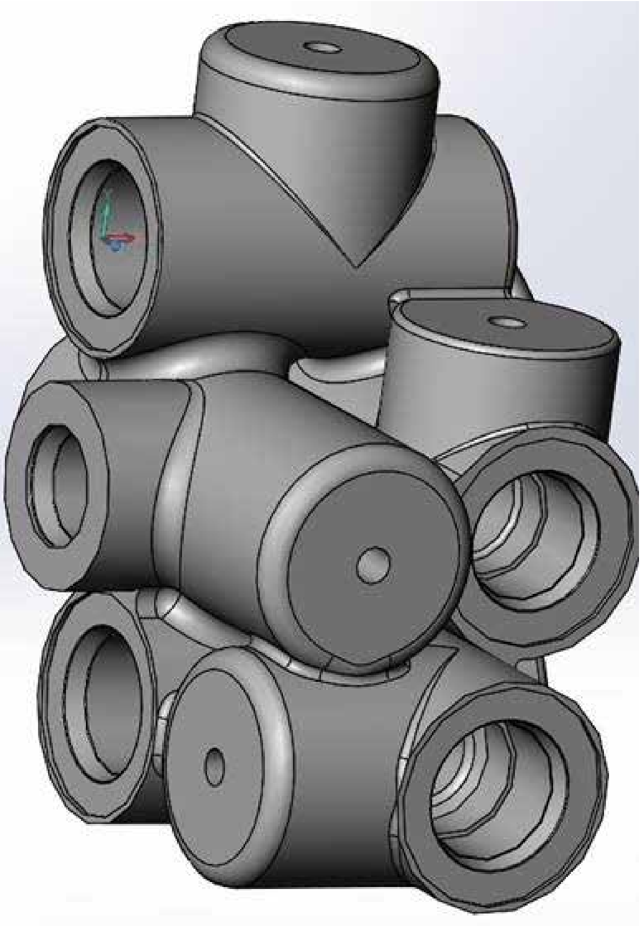
The results from the pressure test show that the durability of the AM printed valve housing as to ultimate pressure is excellent. In the pressure test, a pressure of around six times the operational pressure was achieved without any structural damages to the housing. No fatigue tests have been carried out in this demonstration project.

VALUE / IMPACT

Designing high pressure parts for AM enables new and different application. Hydraulic parts can be more compact enabling placements and applications not previously possible. Additionally, the internal channels can be made much more strait with reduced flow resistance and less pressure loss. This is especially important for continuously operating pumps and valves, where the energy required to drive the system may be reduced significantly. For DT specifically, the new possibilities enable them to launch a new type of product and increasing their potential market.

In certain cases, using AM allows DT to create better solutions for its customer in terms of technical specifications, design and lead times.

The limited budget and time for this project has not allowed for thoroughly testing the AM printed housing, and hence supplementary tests would be necessary to verify the integrity of the valve housing. However, the project has proven that it is possible to prepare design, carry out printing and execute pressure testing of a valve housing within a very short time frame.



Picture 1: Valve housing design

Picture 2: The 3D printed valve housing after glass blasting

Picture 3: The valve housing after testing at approx. 1500 bar

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Duration of project:
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Collaborating companies:
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KNOWLEDGE PARTNERS