FF, Joining of float glass

Project within: FFI, Sustainable production

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FFI

Content

1. Executive summary

Bonding of glass is complex and contains many interacting parts. Not only the adhesive properties and geometry affect the adhesion and thus the joint strength, but also the glass chemistry and surface purity. The glass surface chemistry can vary for many reasons, and this will affect the joint strength. The glass chemistry of the surface is affected by the ingredients used the glass, the manufacturing process, if the glass during manufacturing is contact with the tin bath or the atmosphere in the float glass process, post process like hardening, coating, bending and ageing, etc.

The aim of the project was to allow the introduction of atmospheric plasma treatment for effective glass bonding for the automotive industry with automated contamination detection. Therefore, the overall objective of the project was to create a stable, reliable and verified process to clean and surface activate with atmospheric plasma before bonding glass with and without ceramic frit and in that way replace the primer performed at the glass manufacturer..

For bonding on glass or ceramic frit, the hydroxyl groups, ≡Si-OH on the glass are important. These groups are the reaction sites for silanes in the adhesive and play a very active role in the chemical bonding between the polyurethane adhesive and the glass. The project has studied how the silanoles are affected by different treatments. The area is more complex than what was previously anticipated and it is therefore interesting to work further with the verification with the newly developed method with ESCA /XPS for rapid analyze of silanols degree in the glass surface.

The project has also examined and detected differences in bond ability and surface energy between different glasses, adhesives, aging and plasma treatment. The results from the project show that plasma treatment can clean a glass surface from organic contaminants improve the surface energy from an adhesive bonding point of view and can generate a higher level of cohesive failure. One side effect of the plasma treatment performed in the project is heating of the glass surface. This causes a reduction in the rate of silanols on the surface. To compensate for this, a new plasma process was developed and evaluated by chemical analysis and bonding. The study show good results but it would be interesting to verify the results further with more replicates as well as a process optimization.

As part of quality assurance, the project has worked with contamination detection for mass production. The project has been working to identify possible techniques and then evaluate them with a range of existing equipment on the market. A larger screening was done initially and the equipment that yielded interesting results were further studied in a deeper study with aspect to contamination detection level. Many devices had difficulties in identifying the chosen contaminations on the glass, and of those only a few who were able to determine differences in quantity.

In one of the demonstrator cases a fictitious implementation case was performed where personnel from R & D, from production and technical preparation, and researchers took part. The implementation case was supplemented with a FMEA and business case.

Another demonstrator case was verifying the effect of plasma treatment on coated surfaces and glass before glass bonding. Plasma treatment was compared with other types of relevant pre-treatment methods. A supplementing work on contamination on paint with chosen detection equipments was also performed with different levels of contamination and for an improved understanding in the effect of contamination with glass bonding a study of bonding on different levels of contamination was performed. .

In another demonstrator case one of the automotive manufacturers together with researchers was evaluating the most promising contamination detection equipment in the manufacturing plant for studying the manageability and effects seen with and without washing of the glass.

Dissemination of the project was mainly performed through workshops, training and publications. The project group has been very dedicated and has supplemented each other well so the cooperation and the technical work has generated a lot of knowledge.

2. Background

Bonding of glass is complex and contains many interacting parts. Not only the adhesive properties and geometry affect the adhesion and thus the joint strength, but also the glass chemistry and surface purity. The glass surface chemistry can vary for many reasons, and this will affect the joint strength. The glass chemistry of the surface is affected by the ingredients used the glass, the manufacturing process, if the glass during manufacturing is contact with the tin bath or the atmosphere in the float glass process, post process like hardening, coating, bending and ageing, etc.

Today, these glass surfaces limit the interaction between the joint and the body and the structural load-bearing capacity in the event of a crash. The comprehensive knowledge and capability required to venture to design for this is currently lacking to some extent. In order to achieve this in the future, the process needs to be developed further, to improve the quality and repeatability of the joint between the windscreen and body. This requires deeper knowledge of glass, pretreatment, bonding and methods for inspecting the line in mass production.

3. Objective

The aim of this work was to allow the introduction of atmospheric plasma treatment for effective windscreen bonding for the automotive industry, thus enabling a technology shift where the greater proportion of complex glass and glass designer in the vehicle can be implemented in a safe and secure way with automated contamination detection .

The overall objective of the project was to create a stable, reliable and verified process for cleaning and surface activation with atmospheric plasma before bonding glass with / without ceramic frit. This is to ensure an effective, capable and sustainable process for windscreen bonding that increases the ability to create safe vehicles, allows new innovative design solutions and the increased proportion of glazed surfaces.

Sub-objectives

- increased productivity
- reduced environmental impact
- increased knowledge of glass chemistry and adhesives
- automated contamination detection prior to bonding
- improved development process through inclusion of material suppliers, equipment suppliers and subcontractors (incl. SMEs)
- general knowledge-building for cross-industry outcomes in the Swedish manufacturing industry, including the construction and marine industries
- improved research cooperation, nationally and within Europe, between industry, Glafo and Swerea IVF

With regard to the programme, the project aims to achieve the overall objectives of increased productivity in manufacturing preparation and production processes with a consistent and stable process, also including automated inspection. The project also aims to reduce the environmental impact and contribute to reducing diffuse CO2 emissions, by replacing the current washing and primer processes with plasma, whilst ensuring repeatable, quality-assured adhesive joints, which is a prerequisite for future innovative products that include glass surfaces.

For the more product-related objectives in the 2015-2020 programme, the project aims to improve manufacturing processes and provide increased flexibility, but also to build knowledge that can be used in parallel activities such as improving virtual tools, simulation and optimisation of crash safety, for example.

One sub-objective that also connects to the overall FFI objectives is to improve the research cooperation between the industry, Glafo and Swerea IVF, through shared expertise in the form of material knowledge (glass, adhesive), bonding technology, cleanness, measurement technology and process technology. This will create many new opportunities for cross-pollination of ongoing work and the development of a strong European research network that can connect with other industries, including the construction, electronics and the marine industries..

4. Project realization

The idea of plasma treatment before glass bonding was to clean and pretreat the surface in a controlled manner directly before adhesive bonding. The plan was to replace the primer process and chemicals used at the glass manufacturing and subsequent transport to the automotive companies with the risk of contamination.

High demands are needed on the surface quality of the glass during bonding. Yet there is no industrial method used to measure the joint strength in a non-destructive manner that is quality objective. There are initiatives in this direction, but today you are obliged to create the best possible conditions for the adhesive joint from the start and with a repeatable high quality.

The FF project has had the intention to create a qualitatively repeatable pre-treatment process for bonding of glass and automatic inspection of the glass purity before bonding and thus help to create a total solution for glass bonding. The work in the FF was to investigate the possibility if and to create a method for introduction of atmospheric plasma treatment for effective windscreen bonding for the automotive industry.

The project was divided into seven work packages (WPs). Each WP with its own specific deliverables and milestones. In summary, the project was planning to:

- Build knowledge on the effect of glass and glass chemistry on adhesion when bonding to glass.
- Evaluate the effect of plasma treatment on glass with and without ceramic frit
- Develop process windows for plasma treatment of glass with and without ceramic frit prior to bonding
- Develop guidelines for plasma treatment of glass with and without ceramic frit prior to bonding
- Identify methods for automated contamination detection prior to bonding
- Select one or two automated contamination detection methods for further evaluation
- Test optimized plasma treatment prior to windscreen bonding on one or two demonstrators and evaluate the effect
- Verify atmospheric plasma treatment of the windscreen bonding process, for both lacquer and glass surfaces, in the demonstrator to validate the final effect of the plasma treatment for windscreen bonding
- Prepare for implementation through implementation proposals and production set-up and evaluate the concept in a business case

5. Results and deliverables

The results and deliverables including delivery to FFI goals:

Increased knowledge in glass chemistry and adhesive bonding

The project has studied glass chemistry with different types of chemical analyzes, such as ESCA / XPS, TOF-SIMS, SAC and SEM-EDS and collectively through these methods seen differences between the naked glass frit, glass sides, different batches and suppliers, and verified that the plasma treatment, clean the surface of organic contaminants. The project has also investigated and seen differences in bondability between glasses, different adhesives, how the aging and plasma treatment affects the surface energy and bondability.

For bonding with direct glazing to glass, the hydroxyl groups on the glass are very important, ≡Si-OH. These groups are the reaction sites for the silane in the adhesive and play a very active role in the chemical bonding between the polyurethane adhesive and the glass. The project has seen that the hydroxyl groups are affected by, among other things heat and has verified a rise in temperature of the glass surface by plasma treatment. To compensate for the heating during plasma treatment, the plasma process was further developed and evaluated by chemical analysis and adhesive bonding.

The project area is more complex than what was previously anticipated and the project has therefore not been able to result in a optimized overall process, a new method for the rapid analysis of silanols in the glass surface with ESCA / XPS has been further developed but should be further verified. The project has, however, learned a lot of plasma treatment, glass chemistry and adhesive bonding of glass and has summarized lessons learned and guidelines in the technical report.

Volvo Cars has in a demonstrator case worked with evaluating the new plasma treatment effect on their glasses with different age of the glasses, in a study that would be interesting to repeat for both adhesion and surface chemistry.

Higher productivity, greater flexibility and possible implementation

A specification regarding function and productivity requirements have been developed by one of the automotive companies. The work has been followed up by a fictitious implementation case performed with staff from manufacturing and technical preparation and was supplemented by a FMEA and a business case.

One of automotive companies has in a case worked with the full windscreen bonding ant due to that combined the work performed in the previous PERU project with the work within FF. They have further verified the effect of plasma treatment on paint and glass to verify that plasma treatment is working well on their painted surfaces and glass. The effect of plasma treatment has been compared with other types of relevant pretreatment methods used today or that seems relevant. A deeper chemical study of the difference in the plasma effect seen from bondability point of view among different colors would be interesting to work further with.

Reduced environmental impact

The further developed plasma treatment process has the potential to result in that pretreatment can be made at the automotive companies directly before bonding. To ensure this requires among other things, control of glue, glass surface to be bonded and verification in lab. All adhesives for glass cannot be used with this pretreatment process and various glass surfaces have different improvements with bonding. The advantage when this works is that there is no risk of contamination between pretreatment and bonding during transport (now the coating with primer is performed by the glass supplier) and risk of that the primer becomes old and the glass must be sent back for activation. Another advantage with this plasma process is the reduced amount of needed chemicals. This project has although made no environmental assessment after previously measured good results in the previous project, PERU.

Auto-purity assessment before gluing

A specification for how contamination detection should be performed inline production was developed in a workshop. This was followed by a literature study of possible techniques and complemented with a visit to the fair Part2Clean. Suppliers of equipment based on these techniques were then contacted and a study of different types of equipments was performed. The methods that were proven useful for analysis of the chosen contaminants were selected for a deeper study regarding detectability of different levels of contaminations.

Two automotive manufacturers have evaluated the most promising equipments. One at their manufacturing plant close to cell with windscreen bonding and the other to evaluated the possibility of detecting the corresponding levels on painted surfaces. The later study was supplemented with a correlating bonding study to understand in what levels of contamination that will affect the bonding.

Generic knowledge-building and stronger research collaboration

The project has been working with both dissemination of knowledge and to generate new knowledge. Dissemination of the project was e.g. performed in courses; one in glass and glass chemistry and one in adhesive and bonding of glass. Dissemination of expertise was also created through joint research work, meetings and reports. External dissemination of the project results was performed through publications, see below. Non-confidential parts of the project results can also be disseminated interprofessional through suppliers and research organisations in the form of commissions or other collaborations.

The base for FF has been a research team including staff from the automotive companies, glass supplier, adhesive manufacturer, plasma agent and two research organizations. The selection of project team was very successful for the accumulation of knowledge and the ability to be able to come as far as technical project. Several of the companies have not worked together in previous research projects and the two research institutes have not worked together either before. The project has had its base in the Swedish companies but has received much good help of the international departments of Sekurit Saint gobain, Sika and the Swedish plasma agents plasma supplier.

6. Dissemination and publications

6.1 Knowledge and results dissemination

FF has conducted both internal and external result dissemination.

How has / will the project results be used and disseminated?	Mark with x	Comment
Improve the knowledge in the area	x	The project participants have gained a deeper understanding of glass chemistry, adhesive bonding to glass, plasma treatment of glass and contamination detection
Be used in other technically advanced development projects	x	There is a request to build further on the results in Peru and FF in a continuation project along with manufacturing staff in order to improve the level in the TRL / MRL scale
Be used in product development projects	x	The project group is discussing how the knowledge can be used in a research project and the content in the project. An aim is to work further for implementation.
Introduced on the market		
Be used by investigations / regulatory / licensing / political decisions		

6.2 Publications

The project has written a tecnical report describing the technical work within FF where 11 subreports are included. The subreports are:

Report no 1, Ceramic frit , Sven Karlsson, Swerea IVF
Report no 2, Surface chemistry on glass, Lars Mattson, Volvo GTT, Peter Sundberg, Glafo
Report no 3, Plasma treatment for improved bonding on glass, Åsa Lundevall, Swerea IVF
Report no 4, Detection of contamination on glass surfaces, A literature study, Emma Holmström, Swerea IVF

Report no 5, Parts2Clean, Visit report from Stuttgart June 2015, Pär Andersson, Emma Holmström, Swerea IVF
Report no 6, **Automated contamination detection in automated manufacturing process for bonding**, Pär Andersson, Emma Holmström, Swerea IVF
Report no 7, Evaluation of Adhesion of Windscreen Adhesive on Top Coats, Lina Orbeus, Louise Laurenius, Scania
Report no 8, **Bonding on plasma treatments, bonding on contamination and detection of contamination,** Patrik Sjögren, Mikael Jonasson, Scania
Report no 9, Contamination Detection, Kaveh Tondkar, Volvo GTT
Report no 10, Effect of plasma treatment on glass prior direct glazing bonding, Kerstin Wasmuth, Volvo Cars
Report no 11, Activities at Plasmatreat, Jens Peter Jenzen, Volvo Cars

The project is also until now published in 3 papers/technical journals:

Conference proceeding to, Glass Performance day (GPD), Helsinki, 2015 "The influence of surface composition and plasma treatment on adhesion"
Publication in Ytforum No 3 2016 "Plasmabehandling som förbehandling inför rutlimning"
Publication in Teknik och Tillväxt, No 2 2016 "Plasmabehandling som förbehandling inför rutlimning"

7. Conclusions and future research

The complexity of the area is bigger than assumed before starting this project. However, this project has generated a new method for verification of silanols on the surface that would be interesting to work further with as well with the new type of plasma treatment developed. Interesting would also be continuing with working in contamination detection methods to verify the method further and identify whether there are methods available based upon new hypotheses from the end of the project. It would also be interesting to continue working on taking a higher level at the TRL scale and to a greater extent involve production staff and the staff working with manufacturing preparation.

The project is also planning to during end of 2016 have a plasma workshop more focused for the production staff and after the workshop decide for content in a continuation project or application.

8. Participating parties and contact person

Representatives from the automotive industry, material, equipment suppliers and research institutes have performed the work within the FF project. Partners and representatives can be seen in the table below.

Partners	Contact persons
Swerea IVF	Åsa Lundevall
Glafo	Christina Stålhandske
Volvo Cars	Kerstin Wasmuth
Volvo GTT	Kaveh Tondkar
Scania	Carl Appelgren
Sika	Magnus Fröström
Sekurit Saint Gobain	Jens Altes
Agaria	Jonas Axelsson