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„Improve the aging resistance of adhesive glass bonds by environmentally surface treatment processes“

Abstract

Glass is an ideal material due to its high chemical resistance, its low specific weight as well as its high level of transparency. To accommodate for the material's high brittleness, adhesive joints have increasingly been used in glass construction. In order to realize ageing resistant bonded glass joints it is necessary to introduce environment-friendly, process-integratable procedures for surface activation or for the application of adhesive layers [1]. It has shown, that besides wet chemical and flame pyrolytic procedures (Pyrosil®), atmospheric plasma treatment in combination with plasma polymerization contributes to a higher stability of bonded glass joints.
The current state of knowledge restricted the proposal of a theory on the effect of surface activation on the adhesion qualities of the materials surface. Therefore, the above mentioned procedures were investigated in a research project in terms of their modes of action i.e. bond strength, durability as well as the glass surface.

Experimental part

Surface treatment methods

1.) Application of silane bonding agents

A state-of-the-art wet chemical pre-treatment of the substrate surface for the realisation of non-ageing glass bonds is the application of silane bonding agents. They are chosen to match the polymer, e.g. an amine group as a reactant to an epoxy resin [2].

2.) Pyrosil® pre-treatment

Another surface treatment of glass before adherence is the surface silication by flame pyrolysis (Pyrosil® process). The Pyrosil® process is a flame pyrolytic deposition of an amorphous, highly cross-linked silicate layer. In the process, a substance containing silicon is added into the flame [3].

3.) Pre-treatment, activation and polymerisation with athmospheric plasma

An alternative to the mentioned methods for pre-treatment is the atmospheric plasma process. This physical surface treatment had been used predominantly for the surface activation of metals and plastics. It is possible to generate both a clean surface and chemical surface modifications during surface activation [4].

Results of investigations

Normal float glasses (soda lime glass) as well as the more resistant borofloat glass were included in the tests.

The methodology for a systematic optimisation of glass bonds by combining suitable glass properties with methods for pre-treatment and adhesives initially requires a characterisation of the state of the materials and surfaces prior to the adhering. Surface topography with laser profilometry, contact angle measurement, SEM as well as surface analysis with EDX and SNMS were chosen as suitable and promising methods.

The results of the mechanical tests, the macroscopic evaluation and the surface analysis were correlated. Based on the information gained from the correlation, the different process parameters (intensity, duration, gases, etc.) were adjusted with regard to their effectiveness. By this adjustment, the durability of the glass bonds can be improved. Selected examples illustrate the results of the surface investigations.

Contact angle measurement

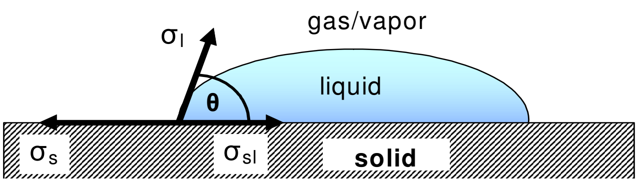
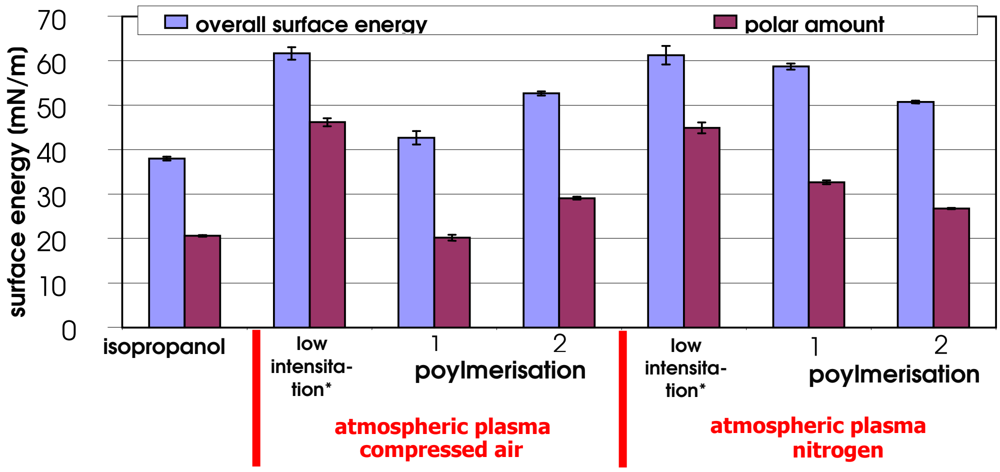


Figure 1: Contact angle of a liquid sample

The elementary method for analysing surface properties is the contact angle measurement (Figure 1). Due to the high surface energies after the plasma pre-treatment, contact angles could only be measured with the test liquids glycerol and diiodmethane.

The contact angle measurements after plasma activation and plasma polymerisation show an increased wettability compared to the pre-treatment with Isopropanol (Figure 2). There is no visible influence on the wettability within different parameter variations (high and low intensity).



*measurement only with diiodmethane und glycerol; demin. water spreads

Figure 2: Results of the contact angle measurement on float glass after plasma activation and plasma polymerisation with compressed air and nitrogen

Surface topography

The influence of the surface treatment on the surface topography was analysed with an optical auto-focus sensor. The structure of the plasma polymer layer could be made visible using the reflexion mode. (Figure 3).

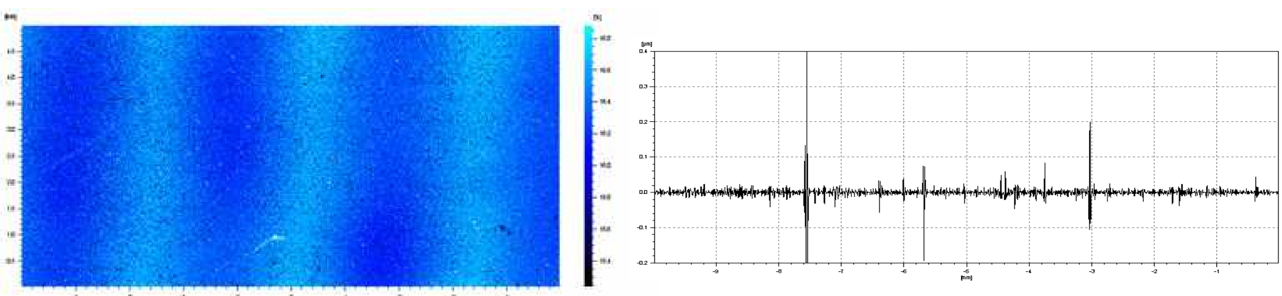


Figure 3: Surface profile after plasma polymerization (the lamellar application of the precursor is visible)

Surface analysis with XPS

The changes of the chemical composition of the glass surface were examined with X-ray photoelectron spectroscopy (XPS). It could be shown, that no organic contaminations were present after the Pyrosil® and atmospheric plasma treatment. By wet-chemically applying silane, employing the Pyrosil process and the AT plasma polymerisation with hexamethyldisiloxane (HMDSO), compact layers could be produced. Alkaline compounds were not detectable. Dependent upon intensity, a reduction of carbon concentration and an increase of oxygen concentration were detected after the atmospheric plasma activation. [5].

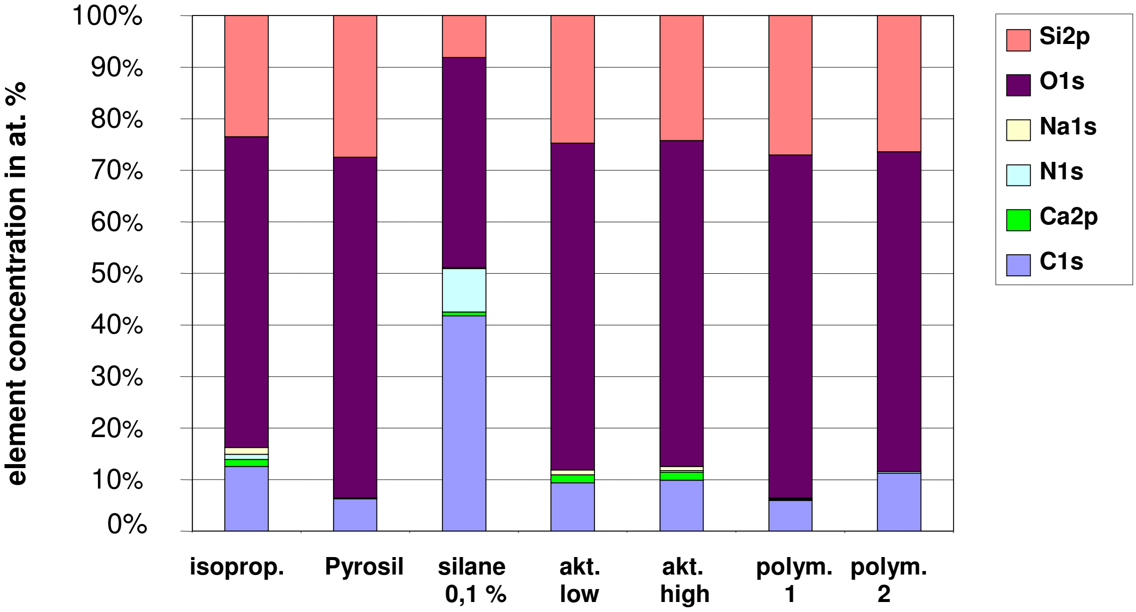


Figure 4: XPS measurements on different pre-treated glass surfaces

Summary

It could be demonstrated that pre-treatment procedures have an influence on both the glass surface and on the used adhesive. This illustrates the basic problem of bonding: pre- treatment methods and adhesive always have to be coordinated since different adhesives can react completely different despite employing identical joining materials and treatments.

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