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J. Schneider/ H. Techen/ R. Stengler

Determination of the Scratch Resistance of Annealed and Tempered Glasses by Using UST (Universal Surface Tester)

Abstract

The use of annealed and thermally tempered soda-lime-silica-glasses in architecture and civil engineering has increased rapidly during the last 15 years. Especially in structural applications, tempered glasses are used more frequently due to their superior mechanical strength compared with annealed glasses. Recently, damages of tempered glasses on the building site by scratching during the cleaning procedure were reported more frequently. Therefore, a more detailed description of the mechanical behavior of glasses subjected to scratching is required for an improvement of the cleaning methods or requirements for special coatings to protect the glass surface.

In this paper, the method of using UST (Universal Surface Tester) for the determination of the scratch resistance of glasses is described. Commercial annealed soda-lime-glasses and tempered glasses made from the same annealed basis glass were tested.

The instrument and its resolution allow a direct analysis of the applied load versus scratch depth and a comparison of the influence of different intenders (diamonds) and their geometries. The results in this paper show that the intender geometry is very influential on the scratch pattern.

1. Introduction

The brittle nature of glass prevented real structural applications of glass in architecture up to the 1990s. Due to improvements in tempering and laminating big sizes of flat glass, today glass is not only used as a cladding material in windows or façades but also as a real structural element. More and more, the glass sustains high and permanent structural loads and even high local stresses are introduced in holes (fig. 1, 2).

Fig. 1: Structural application of tempered glass
(Point fixed glass in-plane loaded, 3 Pacific Place, Hong Kong)

Fig. 2: Structural application of tempered glass
(Glass beams, Lehrter Bahnhof, Berlin)

The predominating glasses used for structural applications are heat-strengthened and tempered glasses laminated together by transparent Poly-Vinyl-Butyral-(PVB)-interlayer (fig. 3) to allow a post-breakage behavior of the glass elements. The permanent compression stress on the surface of thermally strengthened glass (fig. 4) moreover prevents a crack propagation and hence a dependence of the glass strength from the load duration. But also laminated float glasses are now commonly used in engineering in large sizes, e.g. for glasses that prevent from falling.

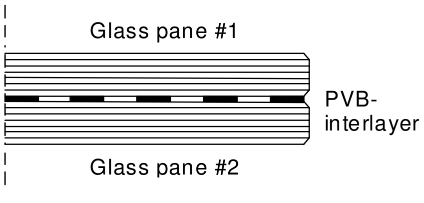


Fig. 3: Principle of laminated glass

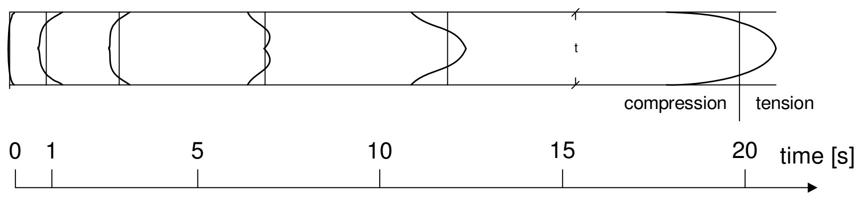


Fig. 4: Stress development during tempering

Recently, damages of tempered glasses on the building site by scratching during the cleaning procedure were reported more frequently. Within the community of façade cleaners and architectural glass specialists [5, 6], it is evident that tempered glasses are somehow more sensitive to scratches than annealed glasses or at least that scratches in tempered glasses are more visible than in annealed glasses. This already led to different cleaning regulations for annealed and tempered glasses [7]. In some projects, façade cleaning companies and their insurances even refuse to clean tempered glasses now due to their “sensitive surface”.

Therefore, a more detailed description of the mechanical behavior of annealed and tempered glasses subjected to scratching is required for an improvement of the cleaning methods or requirements for special coatings to protect the glass surface. Recent publications on the scratch resistance of annealed glasses [2, 3] and a basic work on annealed and tempered glasses from 1999 [1] give evidence that the mechanisms during scratching are not fully understood yet.

2. The principle of the Universal Surface Tester (UST)

The Universal Surface Tester (UST), fig. 5 developed by company *Innowep* in collaboration with *Darmstadt University of Applied Sciences*, determines the micromechanical properties of materials near the surface.

Fig. 5 UST-Universal Surface Tester

Several standardized testing heads or spikes (e.g. different diamonds, steel needles,…) can be used to test various types of surfaces. The procedure (fig. 6) is a scanning along a straight line divided in three steps:

1. Scanning with a minimal load of 0.7mN (determination of the surface profile),
2. Scanning with defined loads (range 1-1000 mN) (determination of the total deformation *G*), the load can be increases by load steps or gradually during the testing,
3. Final scanning with 0.7 mN (recovery of the elastic portion *E*, plastic deformation *P* remains).

The sample itself is fixed on a small table by vacuum and the table itself moves while the testing head remains stable in its position. The resolution of the machine in z-direction (sample thickness direction) is 60 nm.

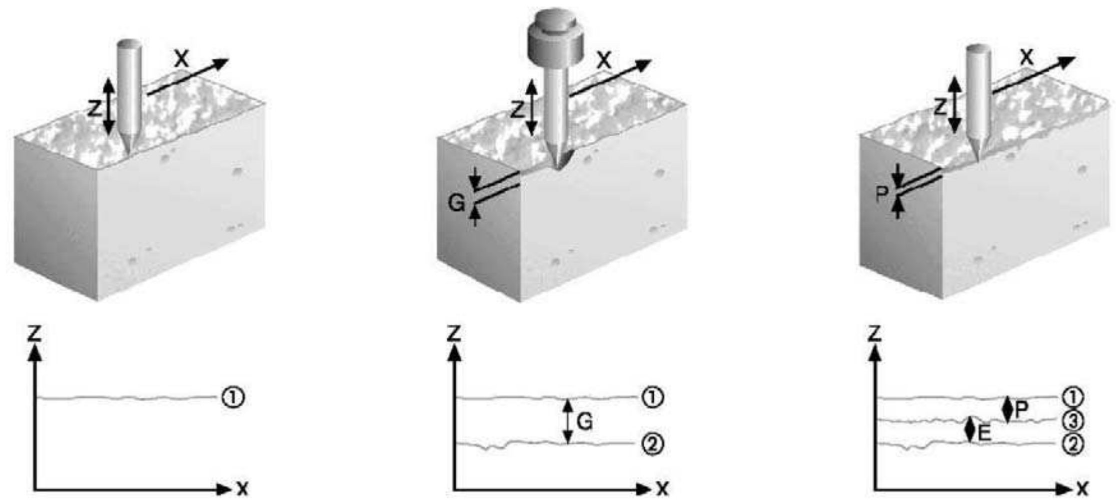
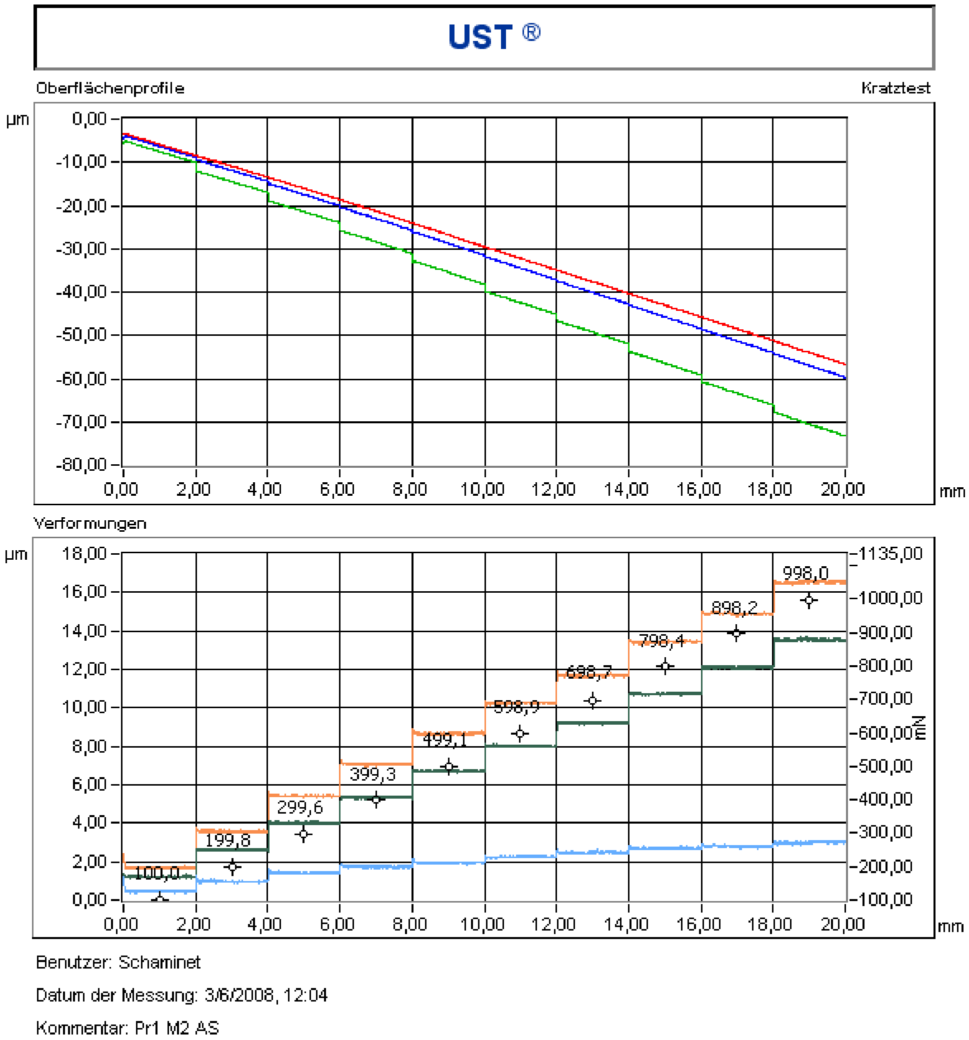


Fig. 6: Procedure of the UST – three step scanning

Figure 7 shows a typical result of a scanning process in the described three steps in two diagrams. Note that the apparent inclination of surface profile (approx. 60 μm on a measuring length of 20 mm = 0,172°) shown in the upper diagram is only due to a slight inclination of the table below the instrument during measurement. From the diagrams it is possible to identify the load and associated deformation in detail. Moreover, any abraded material in the crack during step 2 is identified by step 2 as the graph (blue line) gets irregular in this case.



Upper diagram:

Red line:
Initial surface scanning
(step 1)

Green line:
Scanning the surface while scratching with a defined load. Here, the load increased stepwise by 100mN
(step 2)

Blue line: scanning the surface again without force to determine elastic and plastic deformation
(step 3)

Lower diagram:

Orange line:
Total deformation

Dark green line:
Elastic deformation

Light blue line:
Plastic deformation.

Left axis:
Deformation [µm]

Right axis: Force [mN]

Fig. 7: Resulting diagrams for a measured surface profile and the resulting deformations due to an increased load by steps of 100 mN on a distance of 20 mm with a 60° cone diamond, 8 mm tempered glass

3. Test parameters

General

The goal of the tests described in this paper was to identify the most influential parameters on the scratching behavior. It was assumed from the results in [1, 2] and own experience from glass damages on building site that these are glass composition, load level, loading speed, temper stress and humidity; moreover it was the original intention to look at the influence of tin or fire side from the float process and roller or air side from the tempering process.

To reduce parameters, the role of humidity, the glass composition and loading speed were not studied. The environment was standard climate with 23℃ and 50 ± 10% rel. humidity and the glasses were soda-lime-silica-glasses (float glass) from four different commercial suppliers. The tempered glasses were tempered from the identical float samples in a commercial process. The loading speed was 0,2 mm/s.

Test parameters:

Measuring length on sample:	20 mm (= 2 mm per load step)
Loading speed:	0,2 mm/s
Initial load:	10 mN
Max. Final load:	1000 mN
Load steps:	10 steps with an increase of 100 mN per step
Environment:	23℃, 50%-60% rel. humidity
Glass thickness:	8 mm, 10 mm and 12 mm
Glass type:	Commercial soda-lime-silica-glasses (EN 572) from the float process. The identical glasses were also used for the tempering process. (DIN EN 12150). Glasses were obtained from four different commercial sources.
Glass sizes:	300 mm x 100 mm
Surface compression stress:	3 – 15 MPa (float glass), 90 – 150 MPa (tempered glass)

The surface compression stress of the glasses was measured using the scattered light method with the instrument *Scalp-03* [9]. Note that not only the tempered glasses but also the float glasses had a significant surface compression stress form the float process. Figure 8 shows a typical stress profile of a 12 mm float glass sample with a surface compression stress of approx. 10 MPa.

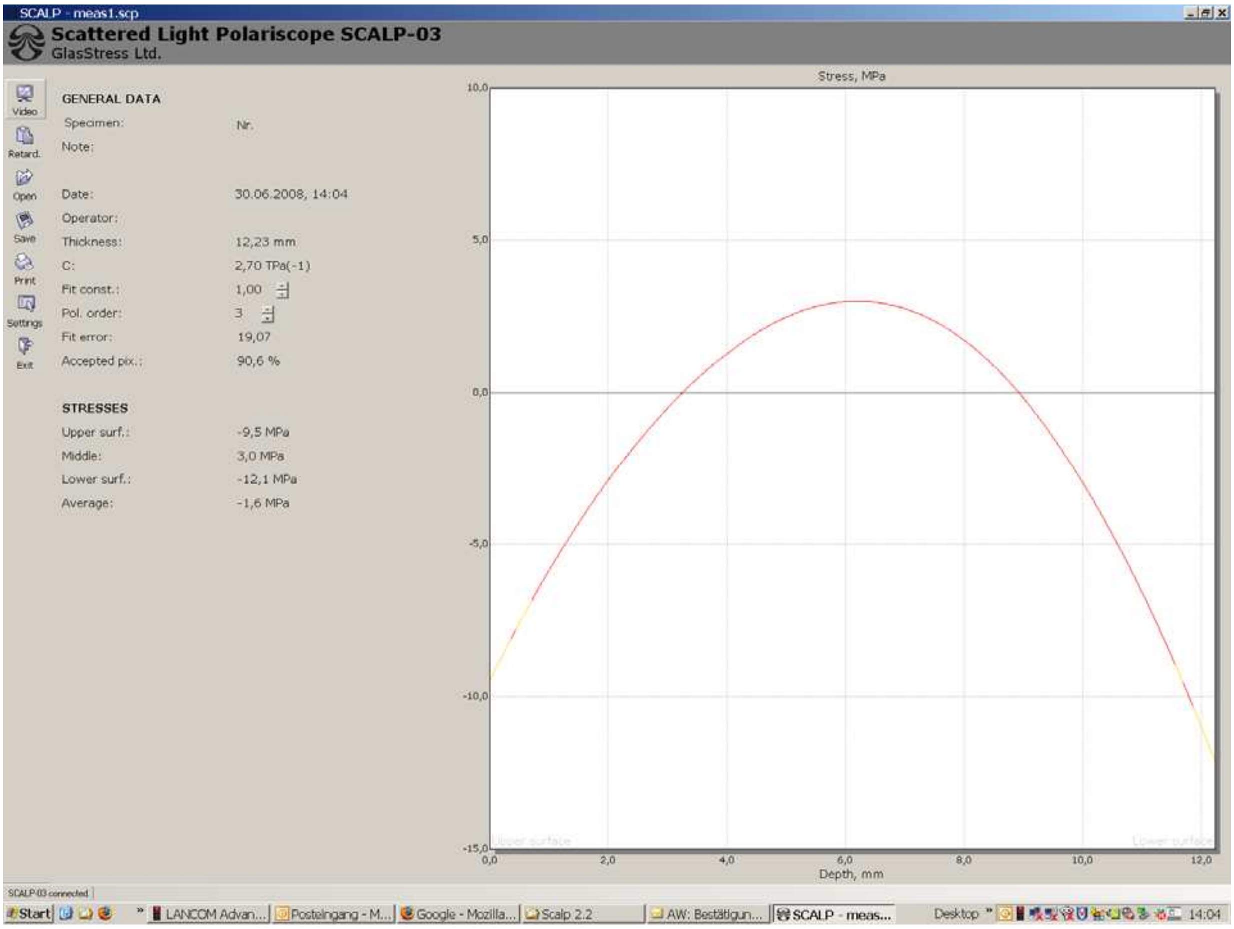


Fig. 8: Stress profile of a 12 mm float glass sample with approx. 10 MPa residual surface compression stress

Intender

In [1, 2, 3], a Vickers indenter was used to scratch the surface and the Vickers intender seems to be generally accepted for scratching tests. As the geometry of the Vickers indenter (diamond, pyramid 136°) is very special, as it is intentionally used to determine the hardness of a material and not its scratch resistance, and as the results from [1] already showed a strong dependence of the results from the degree it had already been used, we decided to use three different types of diamonds instead. This should also take into account that the debris found on glasses on the building site is usually sand, cement, etc. and that it will therefore be important to compare the geometry of the scratching intender with original debris in a later stage. Moreover, the test results (chapter 4) showed that the influence of the intender geometry is more influential than originally assumed.

The first diamond used was a cone type with diameter of 4 mm, 90° tip inclination and a tip radius of 5 μm (fig. 9).

The second diamond used was a cone type with diameter of 4 mm, 60° tip inclination and a tip radius of 5 μm (fig. 9).

The third diamond used was a Ritz type with leading edge 120° tip inclination, a tip radius of 4 μm and a longitudinal inclination of 5° (Fig. 10).

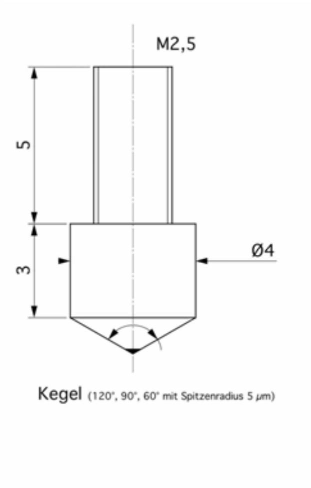
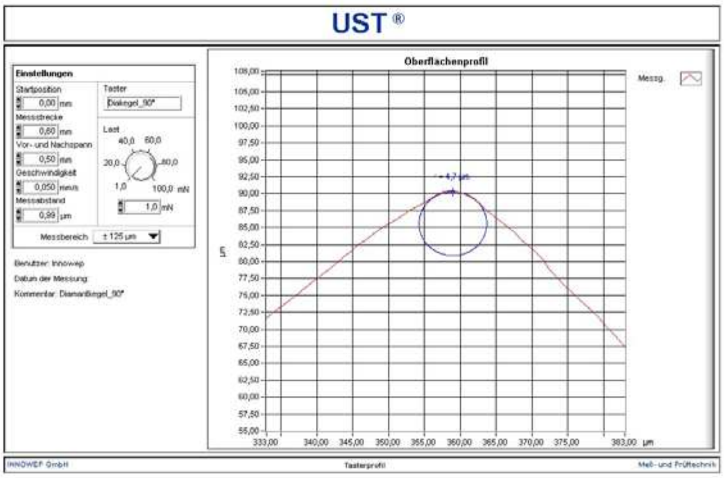


Fig. 9: Cone Diamond with 60° or 90° tip geometry



Cone geometry at the tip measured (r=5 μm)

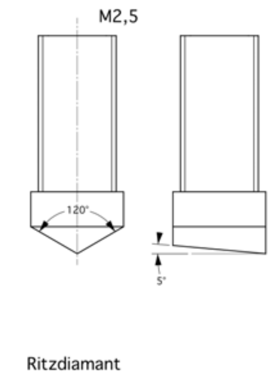
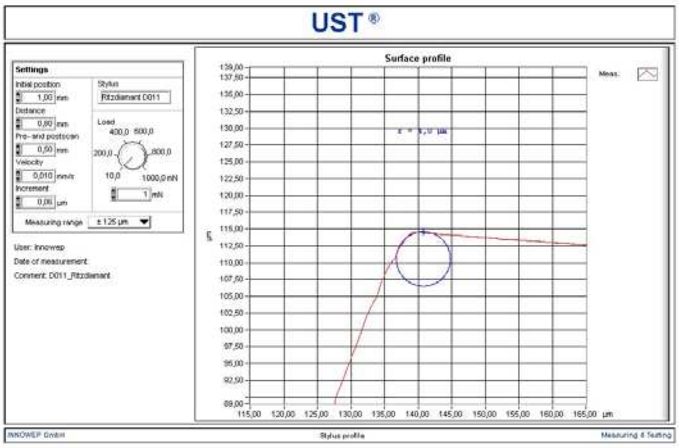


Fig. 10: Ritz-Diamond with 120° leading edge and 5° longitudinal inclination

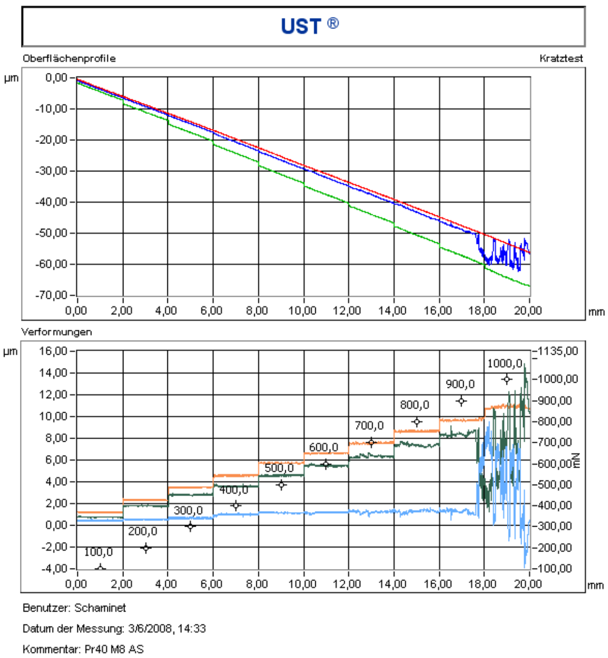


Cone geometry at the tip measured (r=4 μm)

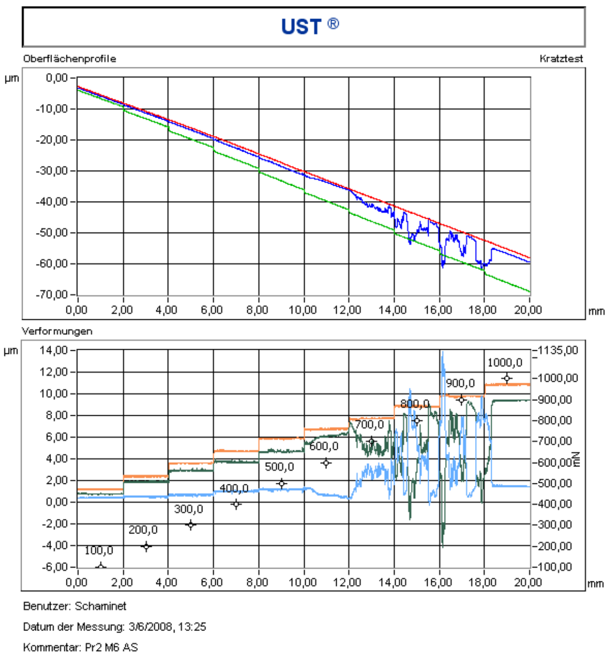
4. Test results

Diamond cone intender with 90° tip geometry

The figures 11 and 12 show typical surface profiles of float glasses in comparison to tempered glasses for a load range of 100 mN to 1000 mN. The total vertical deformation of the intender is almost identical for float glass and tempered glass (≈ 10 µm). This means, there is no significant difference in hardness. But it could be observed by light microscopy that “chipping” [1, 2] in the micro cracking regime starts with vertical forces of 700 mN to 800 mN for float glasses and 300 mN to 400 mN for tempered glasses. By chipping, the apparent width of the crack increases up to 300 µm typically. The width of the cone intender for the total vertical deformation of 10 µm is only $w_{intender}$= 24 µm. This is corroborated by the dark blue line in fig. 10 which shows that glass debris within the crack disturbs the third step of scanning (without force) and causes vertical positive and negative deformations – elastic and plastic deformations cannot be analyzed in this region. This deformations start with earlier on the scratch for tempered glass. Micro-abrasion with debris in small particles on the glass surface could only be observed for float glass and forces above 800 mN (figure 11).

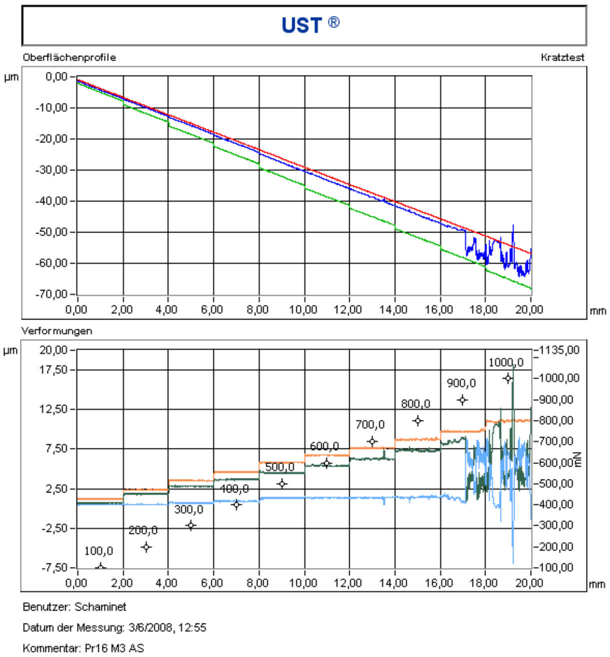


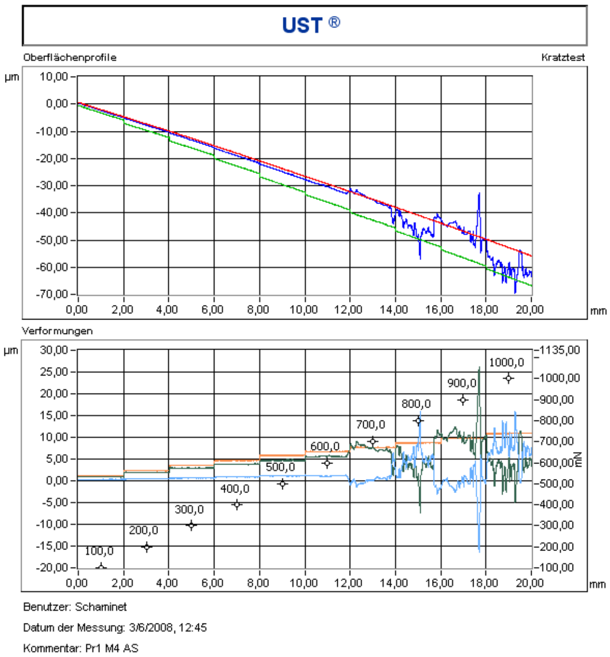
Float glass 12 mm

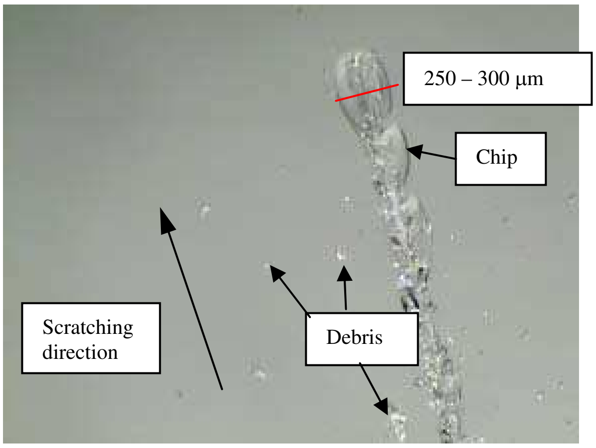


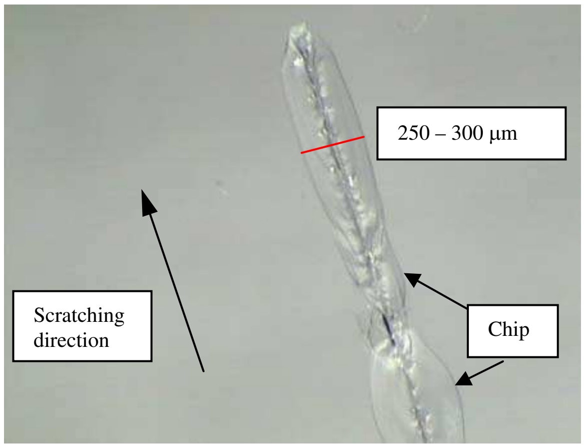
Tempered glass, 12 mm

Fig. 11: Surface profile of a 12 mm float glass sample in comparison with a 12 mm tempered glass sample, 90° cone diamond









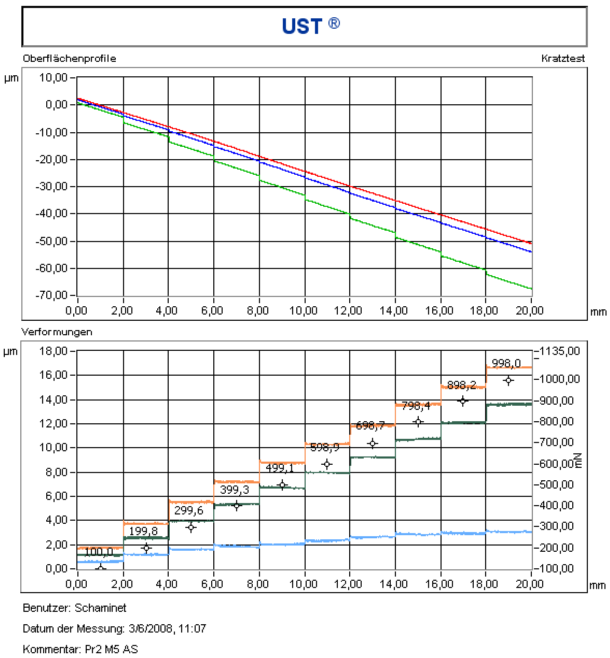
Float glass 10 mm

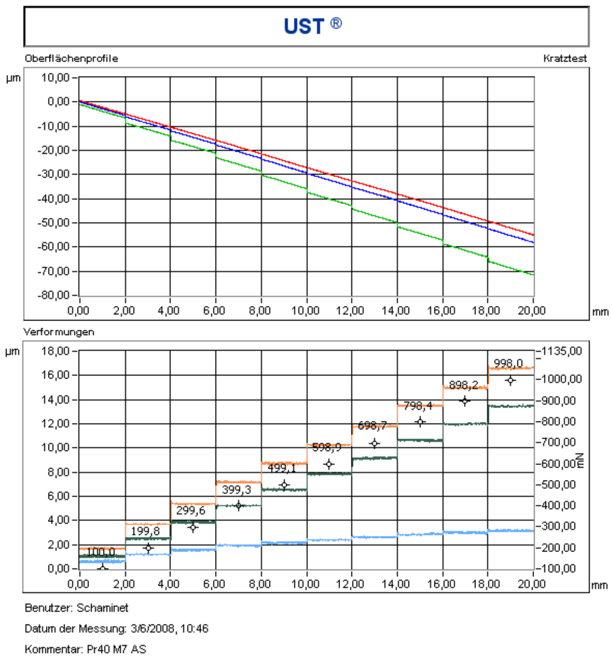
Tempered glass, 8 mm

Fig. 12: Surface profile of a 8 mm float glass sample in comparison with a 8 mm tempered glass sample, 90° cone diamond, light microscopic pictures of the final region of the crack

Diamond cone intender with 60° tip geometry

The figures 13 and 14 show typical surface profiles of float glasses and tempered glasses for a load range of 100 mN to 1000 mN. The vertical deformation of the intender is again almost identical for float glass and tempered glass and increased from 10 μm (90° cone diamond) to approx. 16 μm (60° cone diamond). No chipping could be observed, neither for annealed nor for tempered glass, although the width of the cone intender at the new maximum deformation of 16 μm is almost identical *($w_{intender}$=* 23 μm).

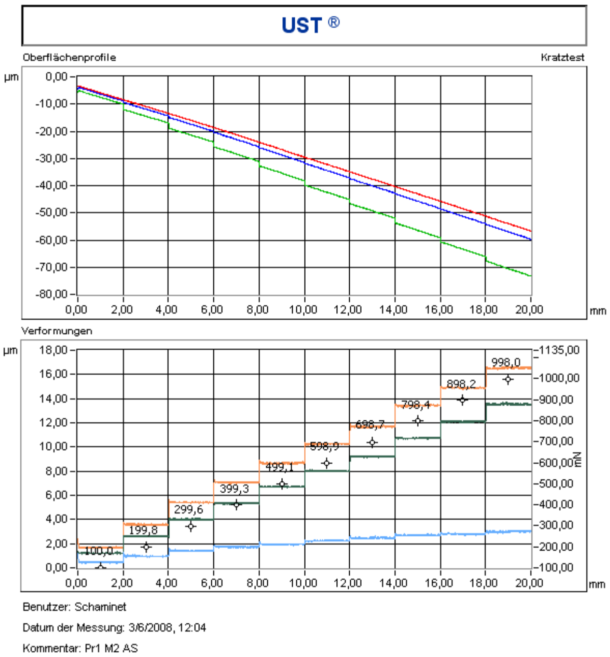




Float glass 12 mm

Tempered glass, 12 mm

Fig. 13: Surface profile of a 12 mm float glass sample in comparison with a 12 mm tempered glass sample, 60° cone diamond



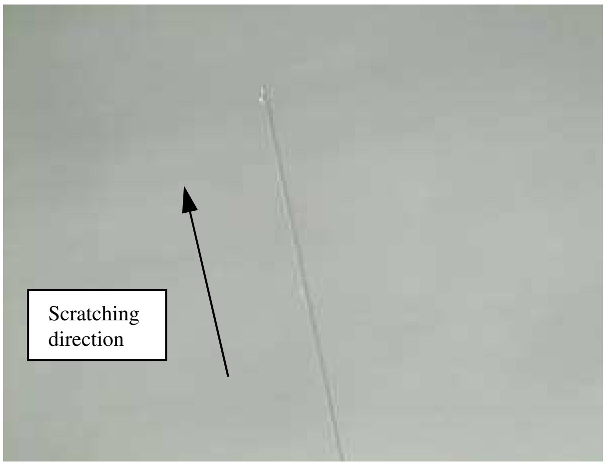
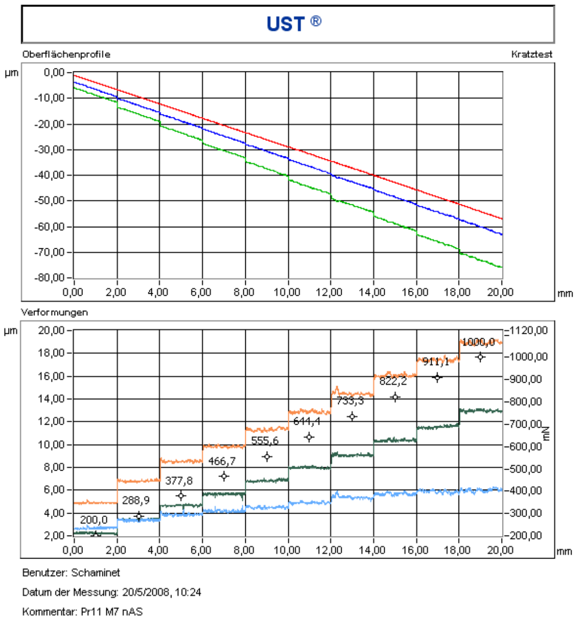


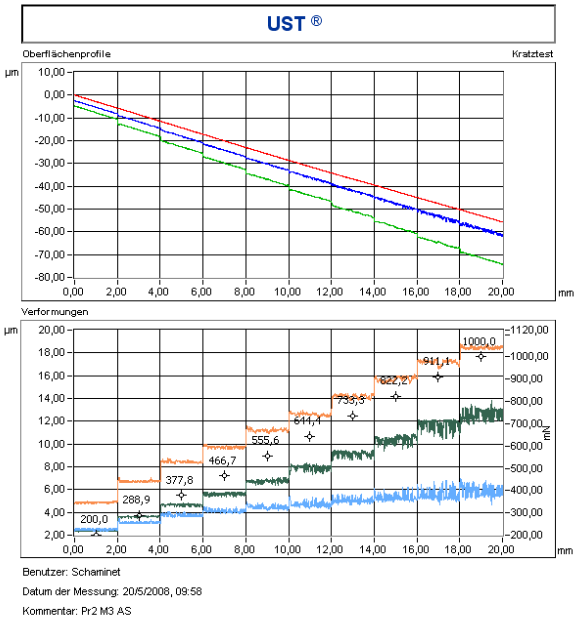
Fig. 14: Surface profile of a 8 mm tempered glass sample, 60° cone diamond, light microscopic pictures of the final region of the crack

Ritz diamond intender

The figure 15 shows a typical surface profile of float glass and tempered glass for a load range of 100 mN to 1000 mN. The vertical deformation of the intender is again almost identical for float glass and tempered glass and increased further from 16 µm (60° cone diamond) to approx. 19 µm (Ritz diamond). Again, no chipping could be observed, neither for annealed nor for tempered glass. The curves remain smooth although slight vertical irregularities can be observed for the elastic and plastic deformations of tempered glass.



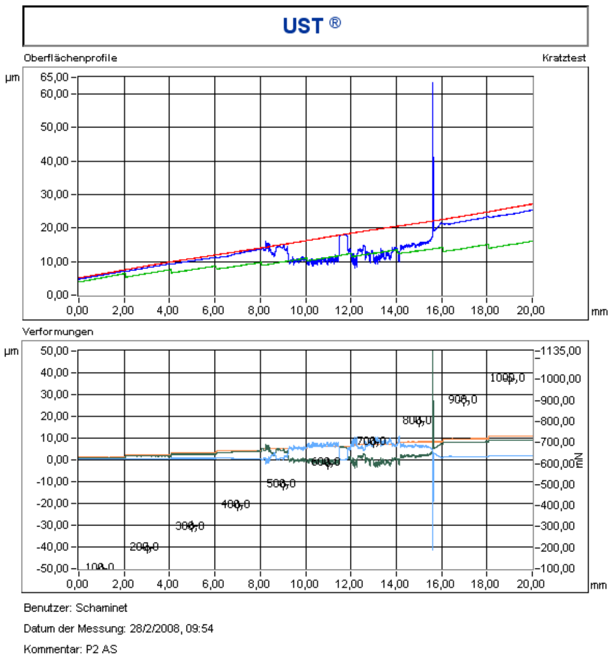
Float glass 12 mm



Tempered glass, 12 mm

Fig. 15: Surface profile of a 12 mm float glass sample in comparison with a 12 mm tempered glass sample, Ritz diamond

To illustrate the great influence even of the quality of the intender, figure 16 shows in contrast a sample of a 12 mm tempered glass tested with an old, already used Ritz diamond. Here, typically long glass “sticks” appear along the crack and chipping starts very early at a vertical force of 100 mN. These sticks could not be observed on float glasses.



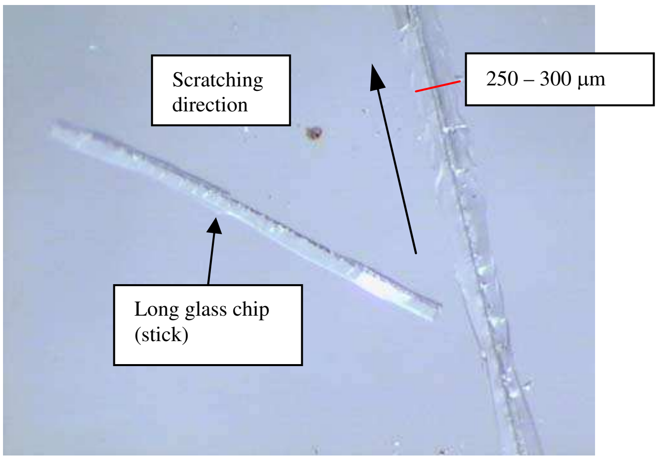


Fig. 16: Surface profile of a 12 mm tempered glass sample, old, already used Ritz diamond, light microscopic pictures of a typical region of the crack

5. Discussion

The test results show that the geometry of the intender and its condition has a great influence on the scratch pattern for the load range of 100 mN to 1000 mN. Generally, this influence was so far much greater than influences from others parameters described in chapter 3. Compared to the results achieved with a Vickers intender [1, 2], especially the micro-cracking regime with chips could only be observed for a diamond cone with 90° angle; the chips did not occur with a slightly "sharper" intenders with 60° angle or a Ritz intender. Contrary, an old, already used Ritz diamond changed the appearance of the crack totally and resulted in very long chips (sticks) for tempered glass.

Generally, if chips occurred, they occurred at a lower vertical force on the intender and to a greater extent for tempered glasses than for float glasses. As the chips "widen" the scratch from 20 to 30 μm (crack widths from intender) to 200 to 300 μm (apparent crack width caused by chips), it is assumed that this effect contributes much to the apparent higher "sensitivity" of tempered glasses to scratching, although the hardness of tempered glass and float glass is not significantly different. If chips occur, scratches just become more visible due to light refractions on the scratches. This corroborates the results from [1].

But as the occurrence of the chips and their form depend strongly on the intender used and its condition, our future research will concentrate on the geometry and form of debris from sand, cement, etc. found on scratched glasses on building sites to compare it to the intender geometries. This helps to identify the intender geometry which results in scratches comparable to those on damaged glass on site and to improve cleaning and protecting techniques.

It is assumed that the loading speed during cleaning could also have an influence as cleaning is done in higher speeds than those in this study and earlier studies [1, 2, 3]. Other parameters from chapter 3 like the amount of temper stress, influence of tin or fire side from the float process or roller or air side from the temper process seem to have smaller influence and will be studied separately.

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