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Non-Destructive Testing of the Strength of Glass by a Non-Linear Ultrasonic method

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The paper presents basis and experimental results of a non-destructive method aimed at determination of the presence of large surface cracks in glass samples by measurements with NAW® technology (Nonlinear Acoustic Wave). The method is based on a transmitted ultrasonic wave in the material from which the non-linear content of the signal can be analysed. A sample containing defects presents nonlinearities in the form of distortions, such as, higher order harmonics that are detected. Nonlinearities in the signal are primarily formed at crack-tips and the number of nonlinearities is proportional to the amount of damage, or defects, in the sample that is investigated. The result of the measurement and evaluation, that only takes a few seconds, is a damage value that is easy to understand and to use for immediate application. A number of preliminary test results and comparisons with destructive testing for various test setups, as well as a recent test strategy including fabricated defects with a nanoindenter will be discussed.

Keywords: Non-destructive testing, Glass strength, Nonlinear acoustic wave

1. Introduction

Glass is a brittle material whose strength is primarily determined by the presence of cracks on the surface (Veer and Rodichev 2011). The strength of the material is limited by the fact that very high stresses arise at the crack tips when the glass is loaded. Without these surface cracks, glass would have a strength that far exceeded many other materials, e.g. steel. Cracks in glass are also of the nature that they grow under load in so-called subcritical crack growth, or stress corrosion (Ciccotti 2009). This means that glass elements loaded for a longer period of time exhibit significantly lower strength than elements that are loaded for a short time. The size and density of surface cracks vary greatly, which results in that the strength of glass exhibit a great variation and that large safety margins must be employed for the strength of the glass when used as a load bearing building material (Veer and Rodichev 2011).

Destructive tests of glass give typical strength values between 30 to 100 MPa and according to measurements referred to in the EU standard EN16612 (2019), the 5%-fractile bending strength (characteristic value) of short-term loaded glass is 45 MPa. At present, there is no non-destructive test methodology to ensure that the glass meets the characteristic strength value specified in standards. This means that there is a small but potential risk that critical load-bearing building components made of glass are of insufficient strength. Moreover, since the strength of glass under load decreases with time, due to subcritical crack growth, it results in uncertainty regarding the load-bearing capacity of existing glass structures.

It has been shown that by use of nonlinear acoustic wave techniques, defects in materials can be detected. In these methods, acoustic waves are transmitted through an object and nonlinear effects, caused by the defects in the material, is analyzed from the signal obtained at the receiver, see for example Donskoy et el. (2001) and Zhang et al. (2013). Nonlinear effects have been observed experimentally for various types of defects, such as cracks, disbondings, delaminations, and other microstructural material defects (Donskoy et al. 2001). In these methods a weighted damage value is obtained for the whole sample based on the number and size of the defects.

The aim of the research is to establish a correlation between nonlinear acoustic wave measurements and the ultimate strength of annealed glass. If such a correlation is established, it would provide a non-destructive strength test method for glass. If successful, the method may be used for quality control and strength grading of glass products as well as process optimization in production of glass. Then glass panes with the most severe surface cracks could be sorted out, with a potential to significantly increase the characteristic strength resulting in that structural glass can be made using lighter and thinner glass. It can also, by extension, affect the process of glass production if one can measure the number and size of surface cracks and minimize them by changing process parameters continuously. Another application of the method may be to measure the damage value in aged glass in existing constructions over time to detect changes due to new imperfections from e.g. wear, vandalism, etc.

Although measurements in previous projects have shown a clear correlation between the size, strength and increasing damage value with duration of applied load (Karlsson et al. 2018), there is a need for further method development and industrial adaptation to ensure a reliable correlation between measured damage value and the strength of glass. More studies are needed to develop the measurement method, but also to develop statistical models for results analysis.

2. Nonlinear Acoustic Wave Measurements

During the last years, various experimental tests including nonlinear acoustic wave measurements and the strength of glass have been performed. The aim of these tests has been to correlate strength of glass with the damage value obtained from the measurements. Conventional acoustic methods of non-destructive testing (NDT) are based on the principles of linear acoustics. These take advantage of detecting effects such as reflection, scattering, transmission, and absorption of acoustic waves. It is then possible to detect cylindrical and spherical holes or other boundaries of the body. Nonlinear ultrasonic methods take advantage of being sensitive to nonlinear scatterers in a medium. Nonlinear scatterers are e.g. cracks, gap contacts and delamination.

In the studies presented here a nonlinear acoustic wave method (NAW®) developed by Haller (2008) is employed. NAW measurements are based on transmitted acoustic waves that are transmitted into the sample via one or more, often ultrasonic, transmitters. A receiver on the other side of the sample detects the wave and the non-linear content of the signal can then be analyzed, see Figure 1a. For a completely undamaged sample object, the received signal will be without distortion and look like the example in Figure 1b, where a sinusoidal signal is transmitted with a frequency and the response from the transmitted signal is the same undistorted signal. For a sample object containing defects, nonlinearities in the form of higher harmonics will be displayed in the received signal, see Figure 1c. Nonlinearities in the signal are strongly linked to cracks and defects in the material and the number of nonlinearities is proportional to the amount of damage or defects in the sample examined. The method is possible to automate since no input from an operator is needed during the measurement and evaluation could be performed with an algorithm. The damage value, which is the result of the evaluation, is easy to understand and easy to use for immediate application, and since the damage value can be saved and documented, it is possible to follow the change in the damage value over time.

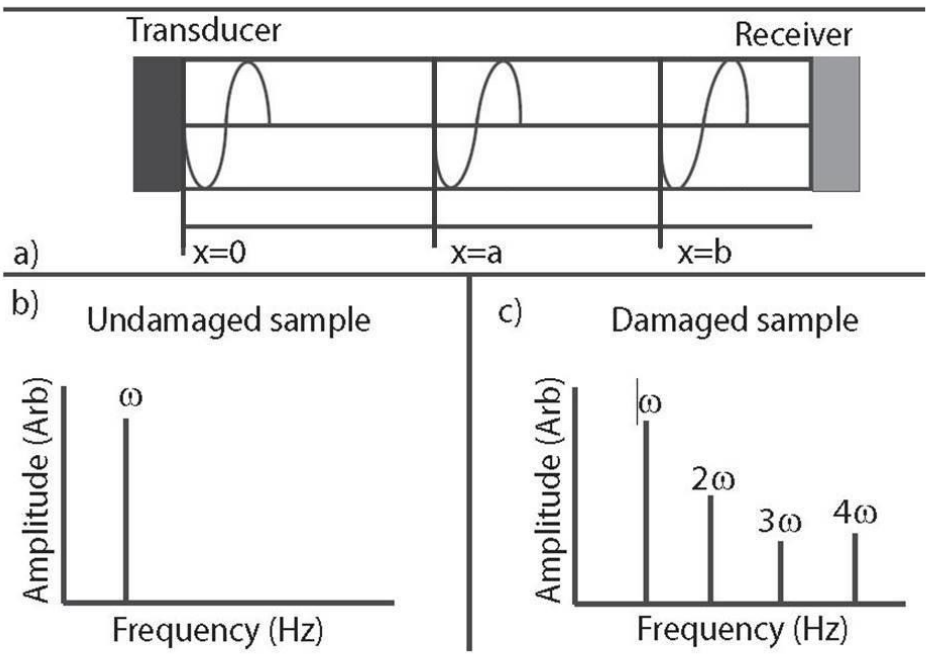

Fig. 1 Schematic sketch of non-linearities showing a) nonlinearities in the propagation of wave at x = b due to a crack at x=a, b) received signal for an undamaged object and c) received signal for a damaged object.

3. Experimental Results

3.1. Preliminary Studies

Glass was examined with the non-destructive test method NAW with simultaneous loading in a 4-point bending test, (Karlsson et al. 2018). The test was done to be0 able to correlate the damage value of the glass, obtained with NAW, with its strength, obtained from a 4-point bending test. Some of the results from that study are presented below. In the study, annealed glass with the dimensions 1000×100×6 mm was tested for three different edge conditions with about 30 test samples in each series. The three edge conditions were seamed, raw cut and flat polished edge, see Figure 2. The test samples were measured with ultrasonic NAW when the glass was simultaneously loaded in a 4-point bending test in both in a setup with a constant load as well as in a hydraulic testing machine, MTS 810. The test with the constant load is shown schematically in Figure 3. In this test only the glass with raw cut edge was tested where a constant load of 20N was first applied and after about one minute the load was increased with an additional 150N. In Figure 4 the result of the test is shown where it is possible to see how the damage value increases with time until failure after about 225 seconds. The increase of the nonlinearities due to crack growth is evident.

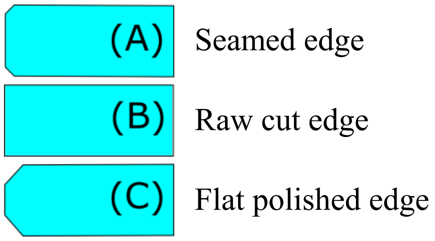

Fig. 2 Edge machining used for NAW measurements and bending testing. (A) seamed edge (swiped) which is rough with defects but the edges are lightly sanded (used before heat strengthening), (B) untreated diamond cut edge, and (C) flat polished edge with matte surface.

In Figure 5, the test arrangement used in the hydraulic testing machine is shown. The glass samples with the three edge conditions were tested at three different loading rates: 0.6 mm/min, 2 mm/min and 5mm/min. Ultrasonic transmitters and receivers were glued to the end edges of the glass's short sides, see Figure 5. A second, low frequency transmitter, was applied on the top of the glass to increase the effect of nonlinear content in the received signal. NAW measurements were carried out at an interval of about 5-10 seconds while the load was increased until the glass failed.

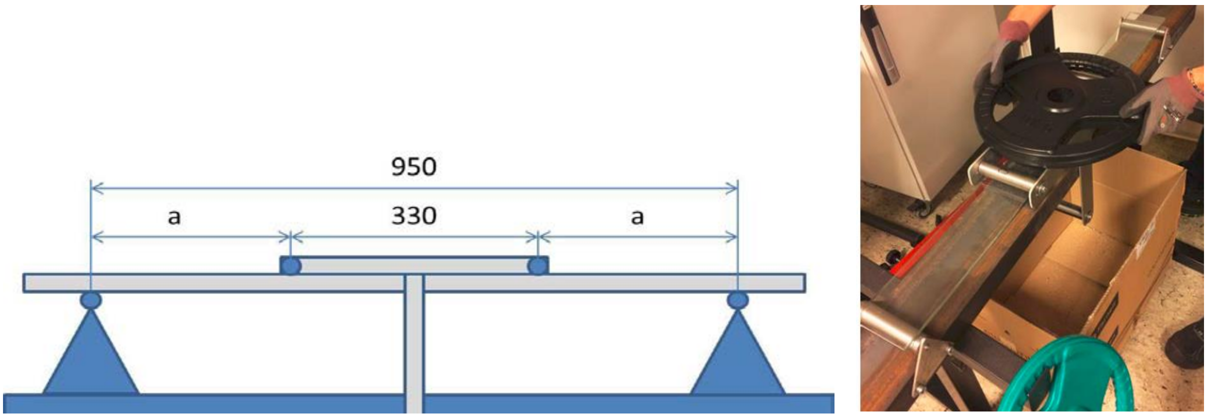

Fig. 3 Schematic drawing and photo of the test setup with the constant load.

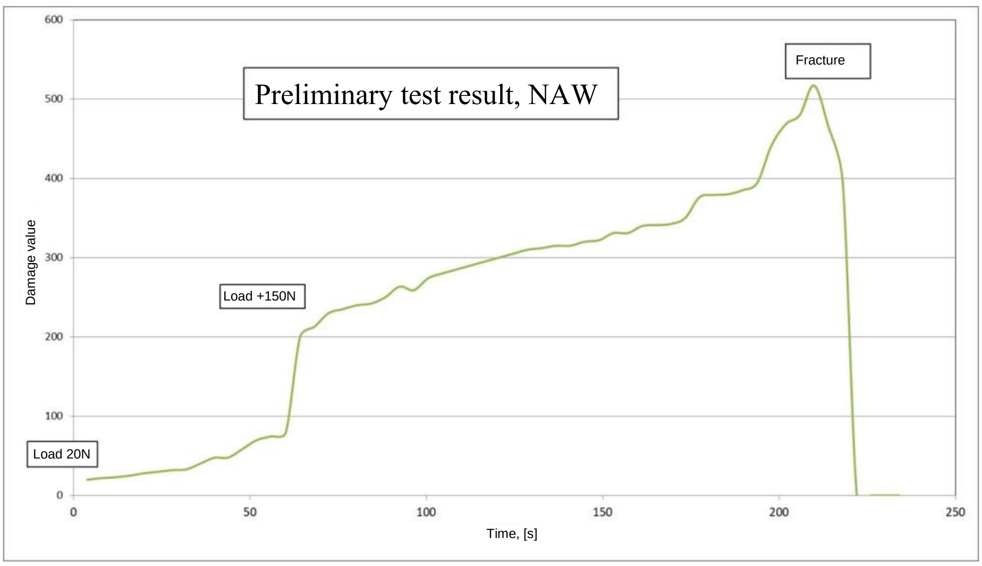

Fig. 4 Results from the test setup with the constant load.

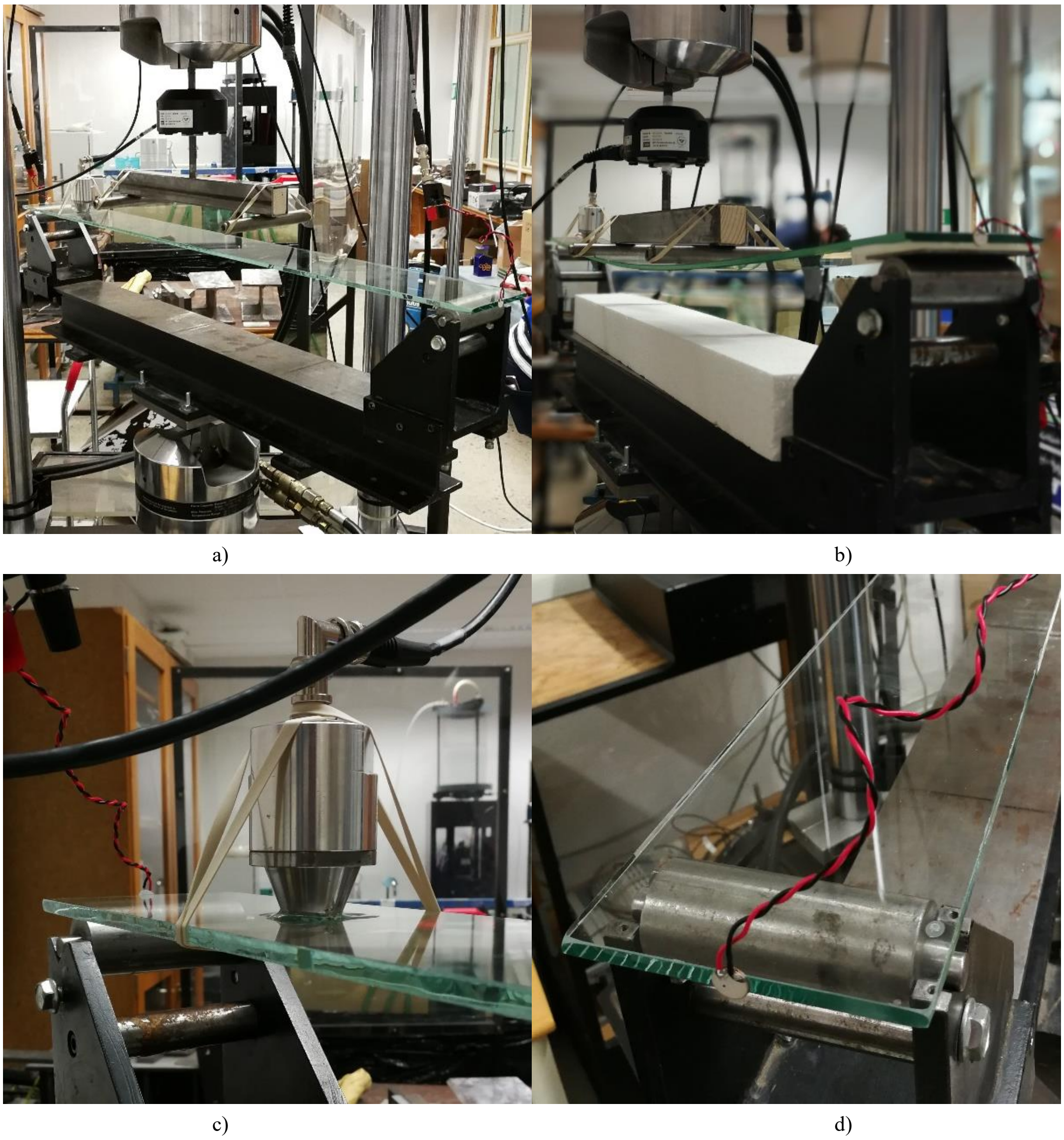

Fig. 5 Non-linear ultrasonic measurement at simultaneous load in test machine showing a) test set-up, b) visible deformations during testing, c) ultrasonic transducer on the edge and a low frequency transmitter on the top of the sample, d) ultrasonic transducer at other end.

Subcritical crack growth occurs in glass when loaded with tensile stresses that exceed a certain threshold value. It was therefore expected that a continuous increase in the damage value until the glass fails would be observed. It is then also possible to correlate the damage value with the strength of the glass since the damage value close to the failure load is detected.

The tests for the three edge conditions were divided into Group A: seamed, Group B: raw cut and Group C: flat polished. The three loading rates of 0.6, 2 and 5 mm/min was denoted by an index to the group name, e.g. $A_{0.6}$, A_2 and A_5. The ultimate load for the three edge conditions at different rates and its standard deviation is shown in Table 1.

Table 1: Ultimate load and standard deviation for the different edge machining and loading rate.

	Edge condition and rate	Average ultimate load [N]	STDEV, ultimate load [N]	No. of samples
Seamed	A0.6	164.3	22.1	6
	A2	168.1	12.7	11
	A5	185.6	21.6	6
Raw cut	B0.6	112.8	3.8	5
	B2	117.2	3.7	14
	B5	126.4	4.6	6
Flat polished	C0.6	181.7	46.0	5
	C2	207.5	41.0	11
	C5	224.4	48.3	6

The evaluation of nonlinearities in the ultrasonic signals was performed by software developed by Acoustic Agree in which the actual damage value was calculated. This was then correlated with the force and deformation measurements from the test machine. There were some problems with noise signals, especially in the first test rounds. These disturbances probably resulted from the test machine, its control systems, computers and other electrical installations nearby. Figure 6 show least square fitted linear curves of the damage value versus the force for all tests within a group and loading rate.

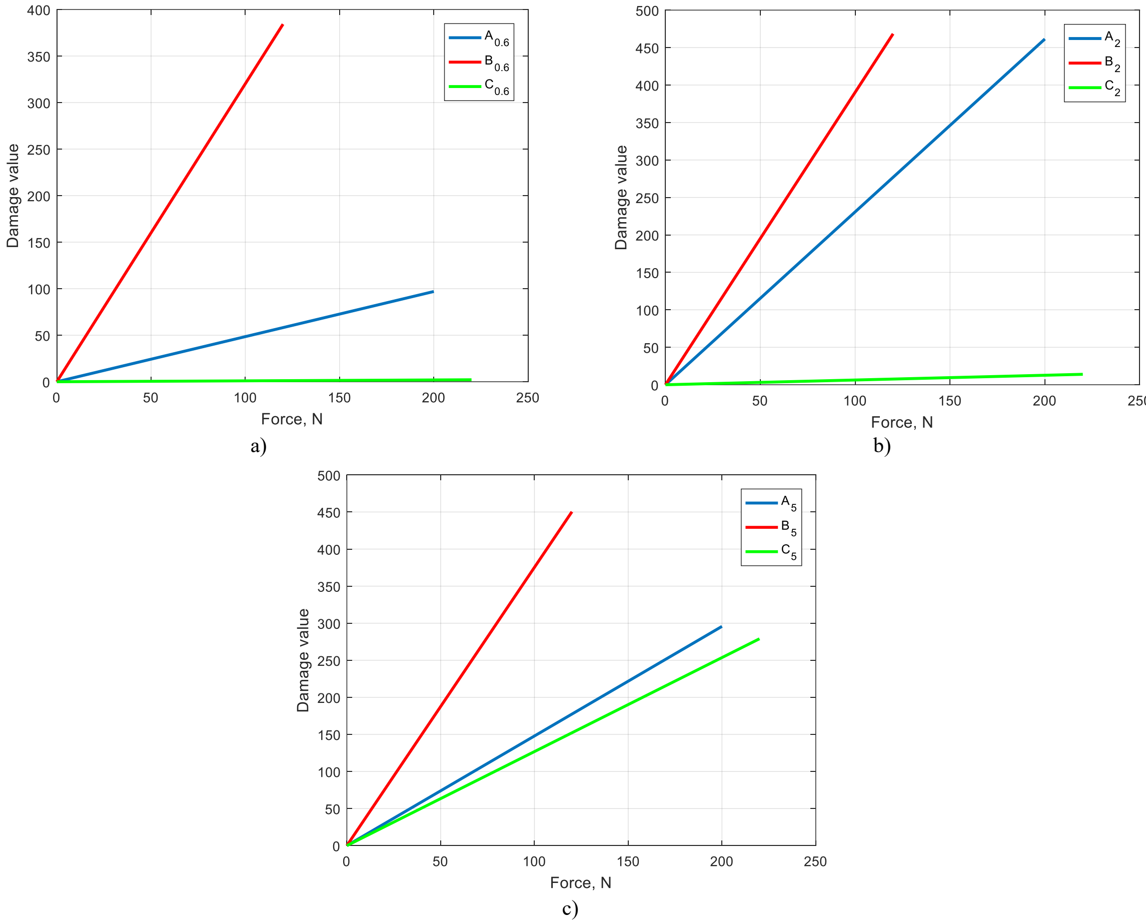

Fig. 6 Linearly fitted curves of damage value vs force for the rates at, a) 0.6 mm/min, b) 2 mm/min and c) 5 mm/min.

In (Karlsson et al. 2018) full results for the NAW tests but also results from tests performed at RISE glass are presented. In Figure 6 it is shown that there is a clear increase in the damage value with increasing load, which was expected. Table 1 shows that the fracture load differs significantly between the three different edge machining. The glass with untreated edge showed almost only half the fracture load compared to the glass with polished edge. It is also clear that the untreated glasses showed lower scattering of fracture load and thus probably have more similar initial cracks. Furthermore, the ultimate failure load is lower for the glass samples that was loaded with lower loading speed, which is an indication of subcritical crack growth. The glass that received the most significant increase in the damage value during the test was the samples in group B with raw cut edge. These glass samples also showed the greatest initial damage value and the lowest strength. The samples with polished edge (group C) showed very small increases in the damage value. This is very interesting as these probably have the smallest initial cracks at the edge and then may not have the same crack growth for an applied load. However, it should be pointed out that the NAW software was mainly calibrated for the samples with a raw cut edge.

Although the measurements in the project showed a clear correlation between the size of the load and the increase in damage value, the majority of questions arose that need to be answered in order to establish a robust non-destructive test method for glass that can be used industrially. Some of these issues that the current project application will examine are listed below:

- Can the signal in the ultrasonic measurements be improved during simultaneous loading in a test machine?
- Can one instead be charged with a constant load to measure the contribution of crack growth to the increase in damage value?
- Is it possible to directly correlate a damage value for a calibrated edge machining to a strength and if so with what accuracy?
- What does crack growth contribute to the increase in damage value with increasing load?

3.2. Present Studies

To overcome some of the problems encountered in the previous studies a new study is currently performed. Therefore, the present project will start with testing in a laboratory environment with low electromagnetic noise in order to establish a solid measurement and evaluation method under controlled forms. In a second stage, measurements will be made in fields on glass fixed in building structures and in production by glass suppliers. In the first study, samples of glass have been prepared at RISE Glass with a mikro- and nanoindenter.

When a sharp diamond indenter using a nano- or microindenter, is forced into apparent brittle materials, these can plastically deform creating permanent imprints in glass (Januchta et al. 2019). At larger loads different types of cracks will also be formed (Arora et al. 1979). The difference between nano- and microindentation is the scale of the indenter tip but also the difference in the force range that you can apply, for nanoindentation 1 mN to 500 mN is applicable while microindentation spans from 0.1N to 30N. The instrumented indentation techniques are commonly used to test the mechanical properties of materials by measuring a force-displacement curve and from that determine the reduced elastic modulus (E_r) via the stiffness, the hardness, the strain-rate sensitivity and more. The indenters typically differ, for nanoindentation a Berkovich tip is conventionally used while for microindentation Vickers indenter is the most common. In Figure 7 examples of imprints on glass is shown. There are also a variety of methods to determine the hardness but the most conventional to use is probably the Oliver and Pharr (1992) method.

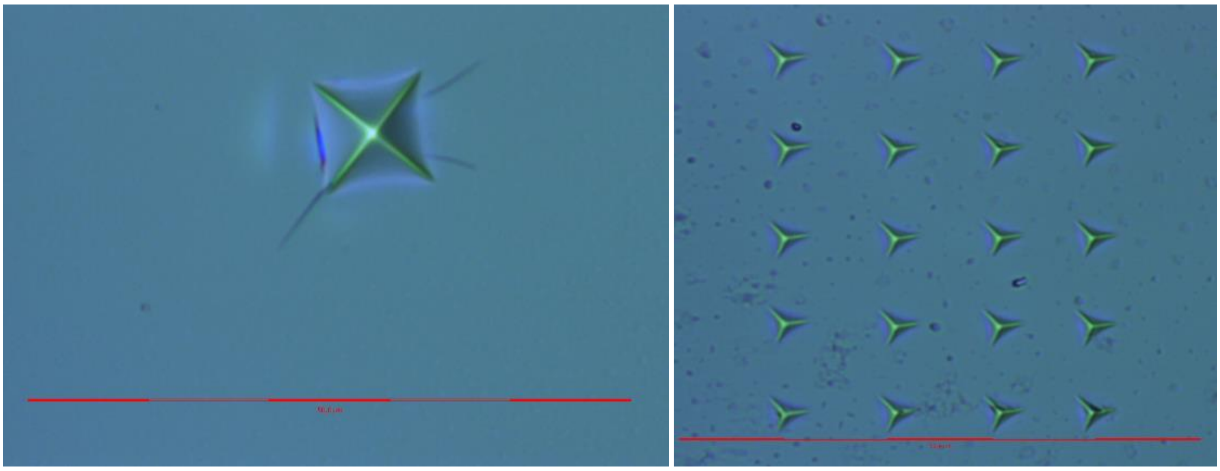

Fig. 7 To the left, example of Vickers indenter imprint in commercial float glass 0.5 N. To the right, example of Berkovich imprints in commercial microscope slide 50 mN.

In a study by Glaesemann et al. (1987) a test with a sharp indenter and the creation of surface defects were performed to be correlated with the strength of glass. A good correlation was found between the strength of the glass and the Vickers indentation crack depth. Since we now have a possibility to perform non-destructive studies, the strength of glass using the NAW technique can be used instead. We will investigate imprinted flat glass by use of nano- and microindentation to study the damage value obtained from the NAW technique and the strength from ring-on-ring test as described in EN 1288-5 (2000).

Five test series are being prepared with 25 samples in each series. The samples that have the dimensions of 100×100×4 mm^3 are being prepared with an indentation at the center of one of the sides of the glass. The test series are produced with various forces where three series are created with a Vickers indenter tip and two series with a Berkovich indenter tip. There will then be five series of samples with varying crack depths together with reference series without any indentations. In addition to the imprinted cracks with the aid of the indenter, other parameters are kept constant (e.g. glass thickness and flat polished edges) to obtain the most reliable result possible.

The prepared samples are then measured using NAW technology to determine the damage value of each sample. To minimize the edge effects on failure load, the samples will be tested to failure in a ring-on-ring test set-up with a support ring of 60 mm in diameter and a load ring of 20 mm in diameter. Analysis of the result of the test and its correlation with the damage value against the load can also be analyzed by use of statistical methods and fracture mechanics theories.

4. Acknowledgements

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