The effect of consolidation treatment on selected mechanical properties of sandstone

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Abstract. An investigation was made into selected mechanical properties of sandstone of Božanov mining site in Czechia using both natural specimens of the sandstone and specimens impregnated with consolidants – liquid products aimed at improving strength and durability of degraded stones. Experiments in three-point bending of notched and cracked specimens made it possible to determine (i) the quasi-static notch toughness as well as the fracture toughness of specimens when they were subjected to static loading, and (ii) the impact fracture toughness and total energy of fracture when specimens were subjected to impact loading. The results of the tests are presented and are discussed with a view to the effects of consolidants.

1. Introduction
Mechanical properties of rock materials have been a subject of interest of investigators for several decades. Among those, which are paid a special attention to, belong fracture properties, namely the fracture toughness. This is a key parameter in rock fracture mechanics used to determine crack initiation and propagation. Its importance can be observed in activities such as the design of rock boring equipment, tunneling, rock cutting and bursting, assessment of stability of jointed rock masses, etc.

Besides this there is also another area where the knowledge of mechanical properties of stones is very important. This area covers conservation and restoration of historical buildings and monuments. The aim of research activities in this area is to improve the resistance of stone elements of buildings and monuments to deterioration and eventual fracture. For this purpose a number of various consolidation solutions have been developed and tested. In this study we have focused on the research of mechanical properties of the sandstone from the Božanov mining site.

2. Quasi-static loading
In order to evaluate the toughness of sandstone at various loading conditions and to consider the effect of various consolidating solutions on the toughness of sandstone we shall start with recalling the results of paper [1]. These results have been concerned with three-point bending of prismatic specimens, nominally $20 \times 20 \times 100$ mm in dimensions with a notch 1 mm wide and 2 mm deep, the span of supports at the tests being 60 mm. The specimens were manufactured from sandstone of Božanov locality, Czech Republic. The stone material can be characterized as a quasi-brittle inelastic silicate composite [2].

Some of the specimens were given a stress cycling treatment to produce a crack at the notch root. Because it was not possible to measure the crack depth during cycling the depth was roughly estimated on the basis of the number of cycles applied. However, this led to a high scatter of crack depths. After...
that the most of the specimens were impregnated with liquid products designed to enhance strength and durability of degraded stone, so called “strengtheners” or “consolidants”. The following products available on the market was used for the treatment: KSE 100, KSE 300, and Paraloid B 72. First two agents produced by the Remmers® company were strengtheners on a silicic acid ester (SAE) base with different silica gel deposition rate. KSE 100 had a low gel deposition rate (10%) in order to prevent excessive surface consolidation and to achieve uniform strength profiles, while KSE 300 was a solvent free product that reaches higher silica gel deposition rate (30%). The last product, Paraloid B 72 manufactured by Rohm and Haas, contained acrylic copolymer based on ethyl methacrylate with methyl acrylate (70:30) and represented a resin widely used in conservation practice for the purpose of consolidation or protective treatment of various materials. Consolidation treatments in the study were performed by partial immersion of stone specimens in liquid consolidant at room temperature for the duration of four hours. This consolidation technique is also known as cold immersion treatment and is the most used treatment in conservation practice. After finishing the immersion treatment, the treated specimens were removed from the bath and the liquid adhered on the stone surface was gently wiped off. Then impregnated stone specimens dwelled for two weeks in laboratory with an environmental condition of 20°C and 60% RH. This period was needed for evaporation of organic solvent (present in KSE 100 and Paraloid B 72) and also for finishing chemical reactions ensuring conversion of SAE to silica gel (KSE 100 and KSE 300).

Mechanical testing was then conducted in such a way that the specimens were subjected to slow monotonic loading with a deflection rate ~ 2.5 μm/s and during the loading the “force – deflection” dependences were recorded. This made it possible to obtain both the maximum force $P_{\text{max}}$ and the absorbed energy to fracture $E_f$. For some specimens we have used the absorbed energy to fracture $E_f$, designated also as a quasi-static fracture energy, to determine the quasi-static notch toughness $KCV^*$, and the maximum force $P_{\text{max}}$ to determine the fracture toughness $K_{IC}$. The relevant results concerning the absorbed energy to fracture are presented in table 1.

**Table 1.** Absorbed energy to fracture in 3PB tests.

| specimen | designation | h x b (mm x mm) | A (mm$^2$) | $E_f$ (mJ) | KCV* (mJ/mm$^2$) | consolid. | note |
|----------|-------------|-----------------|------------|------------|------------------|-----------|------|
| S1       | BO_N        | 18.94x20.66     | 391.3      | 13.27      | 0.034            | no        | notch |
| S2       | BO_20       | 18.21x20.68     | 376.6      | 12.24      | 0.033            | no        | notch |
| S3       | BO_9        | 18.96x21.6      | 409.5      | 8.92       | 0.022            | no        |     |
| S4       | BO_10       | 19.14x21.02     | 402.3      | 8.72       | 0.022            | KSE100    | crack |
| S5       | BO_12       | 6.15x20.65      | 127.0      | 1.93       | 0.015            | Par. B72  | crack |
| S6       | BO_14       | 5.4x20.56       | 111.0      | 1.24       | 0.011            | KSE300    | crack |
| S7       | BO_16       | 19.32x19.18     | 370.6      | 4.24       | 0.011            | KSE300    | crack |

Notes: - Par. B72 in the table is a shortened form for Paraloid B 72
- $E_f$ is a quasi-static fracture energy
- $KCV^*$ is a quasi-static notch toughness ($=E_f / A$)

For a better imagination of toughness properties of sandstone the quasi-static notch toughness $KCV^*$ is plotted for each specimen on figure 1.
Figure 1. Quasi-static notch toughness for the specimens used.

As it is seen from table 1 and figure 1 the highest values of the quasi-static notch toughness $K_{CV^*}$ are found for the specimens S1 and S2 representing a natural untreated material with a notch. This is rather unexpected if we consider the application of various strengtheners as a treatment to improve notch toughness of sandstone material. It is also seen in figure 1 that replacement of a notch by a crack resulted in a decrease in the notch toughness $K_{CV^*}$ by about one third. The results also show that application of KSE 100 product for the cracked specimen S4 had practically no effect on its notch toughness. As compared to the quasi-static notch toughness of specimen S3 the application of Paraloid B 72 for the cracked specimen S5 led to lowering of its notch toughness by more than 30% and the application of KSE 300 for cracked specimens S6 and S7 resulted in a 50% reduction of their quasi-static notch toughness. These results clearly demonstrate a negligible effect of the treatments on the quasi-static notch toughness of sandstone cracked specimens. They also demonstrate a lower quasi-static notch toughness of a sandstone specimen when a notch is replaced by a crack.

It has been already mentioned that in 3PB tests the dependences of force vs. deflection were recorded. For some specimens the maximum force $F_{\text{max}}$ at these plots was used to determine the stress intensity factor $K_I$ characterizing the fracture toughness of a particular specimen. The resulting values of $K_I$ are presented in table 2 together with $K_{CV^*}$ for a comparison to see the effect of consolidators on these quantities. Graphically are the results illustrated in figure 2. Because the numerical values of both quantities differ roughly by an order when using the chosen units, the magnitudes of the fracture toughness in the figure are multiplied by 0.1.

| specimen | $K_{CV^*}$ (mJ/mm$^2$) | $K_I$ (MPam$^{0.5}$) |
|----------|------------------------|---------------------|
| S1       | 0.034                  | 0.331               |
| S3       | 0.022                  | 0.349               |
| S4       | 0.022                  | 0.449               |
| S7       | 0.011                  | 0.561               |

Table 2. Comparison of quasi-static notch toughness and fracture toughness.
Figure 2. Comparison of quasi-static notch toughness and fracture toughness of some specimens.

As can be seen from the figure, the specimen S1 is notched and is untreated with any product; the specimen S3 is cracked and is also untreated; specimens S4 and S7 are cracked and treated with strengtheners: S4 with KSE 100 and S7 with KSE 300. In accordance with figure 1 the quasi-static notch toughness $K_{CV}^*$ decreases when receiving a consolidation treatment. The decrease of $K_{CV}^*$ is greater for the treatment with KSE 300. As it was already stated this decrease makes about 50% of the $K_{CV}^*$ value for the specimen S3.

Exactly different situation is, however, for the fracture toughness $K_{IC}$. If we take the fracture toughness for the specimen S3 as a basis then the use of the product KSE 100 increased the fracture toughness $K_{IC}$ by about 28% and the use of the product KSE 300 by approx. 60%.

3. Dynamic fracture toughness

It is known (see for example [3]) that dynamic rock strengths and dynamic fracture toughness are greater than quasi-static strengths and toughness, and they increase with an increasing strain rate. This means that rock materials have a stronger resistance to fracture in dynamic loading than in quasi-static loading. Because the term ‘dynamic fracture toughness’, can be used in connection with either initiation of the fast crack propagation or with the resistance against propagation of a moving crack, in this contribution we shall use the term “impact fracture toughness” to distinguish between the two. In our previous paper [4] we have reported instrumented Charpy test results as obtained on Božanov sandstone test specimens, it means on specimens of the same material as was that we have used for quasi-static notch toughness tests. Instrumented Charpy impact tester Instron Ceast 9050, used in the tests, provided a single specific loading rate given by its constructional parameters and by the use of a hammer of 1.037 kg in mass. The test configuration of the impact tester provided the striking speed of the hammer $v = 3.8$ m/s and the impact energy $L = 7.5$ J. Dimensions of the specimens were 10 x 10 x 88 mm and the initiation notch was 1.5 mm deep while its width was 1 mm. In the tests the span of supports was 60 mm, i.e. that the span to height ratio was 6. The resulting output of the tests included the total energy of fracture $E_n$ as well as the “force – time” record for each specimen. Knowing the peak forces from these records it was possible to determine impact fracture toughness for the sandstone specimens. Under these conditions we have obtained the impact fracture toughness magnitudes $K_{Id}$. They are presented in table 3. In this study, the product KSE 300 E was used for the treatment of stone specimens. KSE 300 E, similarly to KSE 300, represented the stone strengthenener on a silicic acid ester (KSE) base with silica...
gel deposition rate of about 30%. Compared to KSE 300, the product KSE 300 E contained an elasticized component. Elastic silicic acid esters for consolidation of natural stone were introduced by Remmers® at the end of 1990 and effects of elastic properties of gels with reduced cracking, as compared to traditional treatments without this modification, have still been an object of research [5–7]. The stone specimens to be treated are documented in figure 3. The cracked texture of silica gel created from a non-elasticized type of the strengthener KSE 300 is shown on figure 4 as the gel layer on a dish. After the treatment, thin gel layers cover the stone grains and create bridges between the grains.

![Figure 3. Božanov sandstone specimens prepared for dynamic fracture toughness testing. Scale: white or black square: 1cm².](image)

![Figure 4. Cracked silica gel formed from the strengthener KSE 300 at laboratory conditions (chemical reaction of SAE with atmospheric water vapor, 1 week after pouring the product to Petri dish).](image)
Table 3. Relevant quantities obtained from impact tests.

| specimen number | P (N)  | M (Nmm) | σ (MPa) | \( K_{Id} \) (MPam\(^{0.5}\)) | \( E_n \) (mJ) | consolidant |
|-----------------|--------|---------|---------|------------------|----------------|-------------|
| D1              | 729.7  | 10945   | 65.7    | 4.39             | 109            | no          |
| D2              | 1173.5 | 17602   | 105.6   | 7.06             | 112            | no          |
| D3              | 938.7  | 14080   | 84.5    | 5.65             | 115            | no          |
| D4              | 991.9  | 14878   | 89.3    | 5.97             | 111            | no          |
| D5              | 1037.4 | 15561   | 93.4    | 6.24             | 115            | no          |
| D6              | 631.0  | 9465    | 56.8    | 3.80             | 111            | no          |
| D7              | 658.0  | 9870    | 59.2    | 3.96             | 109            | no          |
| D8              | 1560.8 | 23412   | 140.5   | 9.39             | 135            | KSE 300E    |
| D9              | 1016.8 | 15252   | 91.5    | 6.12             | 123            | KSE 300E    |
| D10             | 1257.2 | 18858   | 113.1   | 7.56             | 128            | KSE 300E    |
| D11             | 1535.2 | 23028   | 138.2   | 9.24             | 136            | KSE 300E    |
| D12             | 1707.1 | 25606   | 153.6   | 10.27            | 140            | KSE 300E    |
| D13             | 1147.3 | 17209   | 103.3   | 6.90             | 132            | KSE 300E    |
| D14             | 1767.4 | 26511   | 159.1   | 10.63            | 145            | KSE 300E    |

Notes:
- \( P \) is the peak force in the “force – time” record
- \( M \) is the bending moment determined as \( P \times s/4 \), where \( s \) is the span between supports
- \( \sigma \) is the bending stress \( = 6 \times M/b/h^2 \) (\( b = h = 10 \text{ mm} \))
- \( K_{Id} \) is the impact fracture toughness \( = \sigma \sqrt{\pi a} F(a/h) \); \( a \) is the crack/notch depth (\( a = 1.5 \text{ mm} \))
- \( F(a/h) \) is a geometric factor; for \( a/h = 1.5/10 = 0.15 \) its magnitude is \( F = 0.97377 \)
- \( E_n \) is the total energy to fracture determined from the “force – time” record

Now we shall make an evaluation of the impact fracture toughness and the total energy to fracture for the group of specimens D1-D7, which were not treated, and also for the group of specimens D8-D14, which were treated with the product KSE 300E.

In order to cover in one diagram not only the absolute values of these quantities but also their statistical parameters we shall use the probability density distribution diagrams for graphical presentation. The probability density distribution diagram (PDDD) for impact fracture toughness is presented on figure 5 for unconsolidated specimens and on figure 6 for consolidated specimens. Their comparison is presented on figure 7.

Sandstone Božanov - unconsolidated specimens

![Figure 5](image-url)  
**Figure 5.** Probability density distribution of impact fracture toughness for unconsolidated specimens.
It can be read from figure 5 that the mean value of the impact fracture toughness $K_{Id}$ is 5.29 MPam$^{0.5}$ and the standard deviation $s = 1.25$ MPam$^{0.5}$. As follows from figure 6, application of the consolidant KSE 300E results in an increased mean value of the impact fracture toughness $K_{Id}$ from 5.29 MPam$^{0.5}$ to 8.59 MPam$^{0.5}$. It is more than a 60% increase. This is accompanied with an increase in the standard deviation by almost 40%. The situation is digestedly illustrated on figure 7.

Now we can make a graphical representation of the total energy of fracture for consolidated and unconsolidated specimens. Alike in procedure taken for illustration of impact fracture toughness we shall plot the PDDDs for the total energy of fracture for consolidated and unconsolidated specimens. figure 8 represents the PDDD for the unconsolidated specimens and figure 9 represents that for the consolidated specimens. A comparison of these PDDDs is made on figure 10.
Figure 8. Probability density distribution of the total energy of fracture for unconsolidated specimens.

Figure 9. Probability density distribution of the total energy of fracture for consolidated specimens.
Figure 10. Comparison of probability density distribution of total energy of fracture for consolidated and unconsolidated specimens.

As it follows from table 3 and figures 8 through 10 the mean value of the total energy of fracture for unconsolidated specimens is 111.71 mJ while that for consolidated specimens is 134.14 mJ, it means it is increased by 20%. However, the standard deviation for consolidated specimens is increased from 2.5 mJ to 7.34 mJ, accordingly by a multiple of almost 3. Owing to this the PDD curve for unconsolidated specimens is rather narrow and that for consolidated specimens is wide. The peak probability of occurrence of the mean value of the total energy of fracture is 0.16 for unconsolidated specimens and 0.054 for consolidated specimens.

In fine it can be said that consolidation treatment of sandstone specimens leads to a certain improvement of both the impact fracture toughness and the total energy of fracture. On the other hand, it increases the scatter of these properties. While the increase of the scatter of impact fracture toughness is less than 40%, the scatter of total energy of fracture is almost 200%.

4. Conclusions
The results of the experimental study proved a beneficial effect of the treatments on the static and dynamic fracture toughness of sandstone specimens as well as on the total energy of fracture upon impact loading of cracked specimens. Concerning the static fracture toughness of sandstone specimens, the highest improvement was reached when KSE 300 strengthener was applied. This type of a consolidant was also used to improve the impact fracture toughness and the total energy of fracture in impact tests. However, the improvement of these quantities was accompanied by an increased scatter. The only negative effect of the consolidation treatment was connected with the quasi-static notch toughness. This result opposes the simple image of a positive effect of the treatment on mechanical properties of sandstone. There is, therefore, a need for a large-scale investigation of how exactly a consolidant works upon a quasi-static loading and why is there a different effect of this loading on the fracture toughness and the quasi-static notch toughness.

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