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# Effect of Chemical Solutions on the Strength of Gosford Sandstone

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**Summary** Chemical alteration of the strength of Gosford sandstone has been investigated to establish the fundamental knowledge for chemically assisted drilling and fracturing, and to evaluate the effect of chemical solutions on the strength, together with acoustic emission measurement during the experiments. The tensile strength varied with the concentration of chemical additives, and became the lowest at certain concentrations which are consistent with the zero zeta-potential concentrations. The maximum strength reduction relative to the water-saturated specimen in  $\text{AlCl}_3$ , PEO and DTAB solutions are 30.8 %, 33 %, and 23 %, respectively. AE activity is the most active in dry specimen, and the least AE activity is found in the specimen saturated with chemical solution. The triaxial strength did not vary significantly with the concentration of the chemical solutions. In case of triaxial compression test where failure mode is dominated by shear failure, there is little effect of chemical solutions on the triaxial compressive strength.

## 1. INTRODUCTION

The role of the chemical environment in rock fracturing has long been considered to be of moderate importance, and chemically assisted crack growth has been investigated by a number of researchers for a long time (for example, Charles, 1959; Brace et al., 1966; Macmillan et al, 1975; Anderson, 1977; Ishido and Nishizawa, 1984). In the area of rock drilling, since Reh binder et al. (1948) reported the use of chemical additives in drilling fluids to enhance the drillability, many studies have been made of various chemicals on drilling behaviour of various materials.

The adsorption-induced reduction in hardness is known as the Reh binder effect, yet it is now suspected that there are several different mechanisms involved in this effect. Reh binder et al. (1948) proposed that the material hardness is altered by a change of free surface energy of the developing crack, due to the adsorption of chemical additives. Westwood's work on the Reh binder effect of several inorganic non-metals (Westwood et al., 1967; Westwood and Goldheim, 1968; Westwood and Macmillan, 1973; Westwood et al., 1974) have revealed that the reduction in hardness has a strong correlation with zeta potential which is an electric potential produced across solid-liquid interface. The zeta potential correlation has also been found for the rate of drilling of quartz crystals,

Westerly granite and quartzite; the drilling speed is the greatest when the drilling with a diamond bit is made at zero zeta-potential. While these theories have been supported by various researchers, controversy still exists on the application of these concepts to rock fracturing.

In the present study, chemical alteration of the strength of sandstone has been investigated to establish the fundamental knowledge for chemically assisted drilling and fracturing. If the rock strength can be chemically lowered intentionally, this technology would be useful to enhance the efficiency of drilling and fracturing. The purposes of the present study are to evaluate the effect of chemical solutions on the strength, and to understand the underlying mechanism of chemically assisted fracturing. Brazilian tests and multi-stage triaxial tests were performed on Gosford sandstone saturated with three different types of chemical solutions. Most of the tests were conducted with the measurement of acoustic emission (AE) in order to elucidate the effect of chemicals on the microfracturing during the tests. In the present paper, a variation of the strength and acoustic emission behaviour with the concentration of three different types of chemicals is reported. The correlation between the strength change and zeta-potential is also discussed.

## 2. EXPERIMENTAL PROCEDURE

A series of Brazilian tests and multi-stage triaxial tests were conducted on Gosford sandstone from NSW, Australia. Gosford sandstone has previously been used for the experiments by Edmond and Paterson (1972), Ord et al. (1991), and Cox and Meredith (1993). It comprises mostly of quartz and quartzite grains together with fine-grained clay and mica material.

The specimens were disks of 44 mm diameter and 21 mm thickness for Brazilian tests, and cylinders of 44 mm diameter and 100 mm length were used for triaxial tests. After shaping, all specimens were dried at a temperature of 110 °C for 48 hours, degassed in a vacuum of  $10^{-2}$  torr at room temperature for about 50 hours and then immersed and placed in a chemical solution of controlled composition until required for testing.

To evaluate the effects of chemical environment on the strength (tensile and triaxial), three kinds of aqueous solution; aluminium chloride ( $\text{AlCl}_3$ ), polyethylene oxide (PEO), and dodecyl trimethyl ammonium bromide ( $\text{C}_{15}\text{H}_{34}\text{NBr}$ , abbreviated hereafter as DTAB) were prepared. The negative zeta-potential exhibited by rocks (Ishido and Mizutani, 1981) in water can be increased to zero or positive values by the addition of various concentrations of the above chemical species to water. Variation in the zeta-potential of quartz or Westerly granite in DTAB solution as a function of its concentration is reported by Macmillan et al. (1975). Zeta-potential's variation of quartz or quartzite in PEO and  $\text{AlCl}_3$  solutions are reported by the US Bureau of Mines.

In Brazilian tests, the specimens saturated with the chemical solutions were loaded inside the vessel filled with chemical solution in order to prevent the solutions from vapourising during the experiments. In triaxial tests, however, the specimen was inserted into the Hoek-type triaxial cell and then loaded under servo-control by averaging signals from three LVDTs between the platens. Axial force was measured by using a strain gauge type load cell. The loading rate was 50  $\mu\text{m}/\text{min}$  for Brazilian tests and 100  $\mu\text{m}/\text{min}$  for multi-stage triaxial tests.

Two AE sensors were attached to the surface of the specimen by electron wax in Brazilian test. One of the sensors (NF-AE-901S) was sensitive to lower frequency range (50 - 200 kHz) and the other one (PAC nano-30) was sensitive to higher frequency range (200 - 500 kHz). In multi-stage triaxial tests, however, only the higher frequency AE sensor was used. It was mounted on the end piece, not only because it was impossible to put the

sensor directly on the sample set up in the triaxial cell, but because lower frequency noises less than 100 kHz due to the high hydraulic pressure, which is required to apply the high load to the specimen in triaxial tests, could not be effectively eliminated in the present system.

The AE instrument, MISTRAS-2001 system, was employed in the tests. Signals from the AE sensors were amplified by a pre-amplifier (Gain: 40 dB, frequency filter: 50 - 1200 kHz) and a post amplifier inside the system (Gain: 20 dB). Event amplitude threshold was set in the screen set-up menu of the test running code. The dynamic range between this threshold and the maximum amplitude recorded was about 50 to 60 dB. All AE signals were described in terms of their counts, energy, amplitude, rise time and duration in the record.

## 3. EXPERIMENTAL RESULTS

### 3.1 Tensile Strength

Figure 1 shows the variation of the tensile strength with concentration of  $\text{AlCl}_3$  solution. The tests in  $\text{AlCl}_3$  were conducted in thirteen different concentrations between  $1 \times 10^{-7}$  mol/l to  $1 \times 10^{-1}$  mol/l. Each strength value plotted in the figure is the average value of 10 to 15 test results. The tensile strength of sandstone is the lowest at concentration of  $2.5 \times 10^{-7}$  mol/l; 30.8 % reduction in strength compared to its strength in water. For Sioux Quartzite in  $\text{AlCl}_3$  solution, the concentration at which zeta potential is zero (ZPC) is about  $3 \times 10^{-7}$  mol/l (Watson and Tuzinski, 1989). The  $\text{AlCl}_3$  concentration giving the lowest tensile strength is close to ZPC concentration for quartzite in  $\text{AlCl}_3$ . It is also found that the strength is almost independent of the sign of the zeta potential.

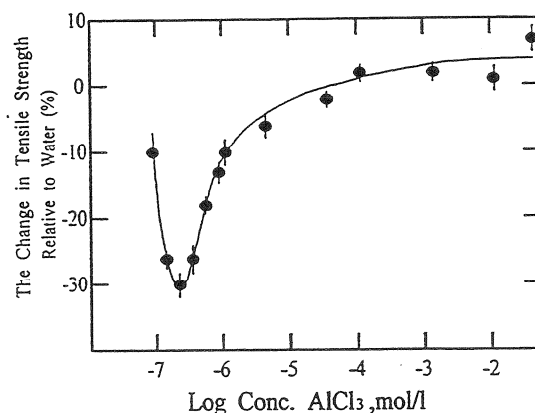


Figure 1. Variation in the change of tensile strength relative to water in aqueous aluminium chloride environments. Note that tensile strength becomes lowest at the concentration of  $2.5 \times 10^{-7}$  mol/l.

In Figure 2, details of the variation of tensile strength with PEO solutions, together with the change of zeta-potential results of Sioux Quartzite in PEO solution measured by U.S. Bureau of Mines (Watson and Tuzinski, 1989) are given. The tests were conducted in six different PEO concentrations between 1 ppm and 100 ppm. In this series of tests, the tensile strength of sandstone became lowest in PEO concentrations range 20 ppm to 100 ppm; 33 % reduction compared to its strength in water. These concentrations giving lowest tensile strength agree well with ZPC concentrations for quartzite in PEO solutions, namely, 15 ppm to 100 ppm.

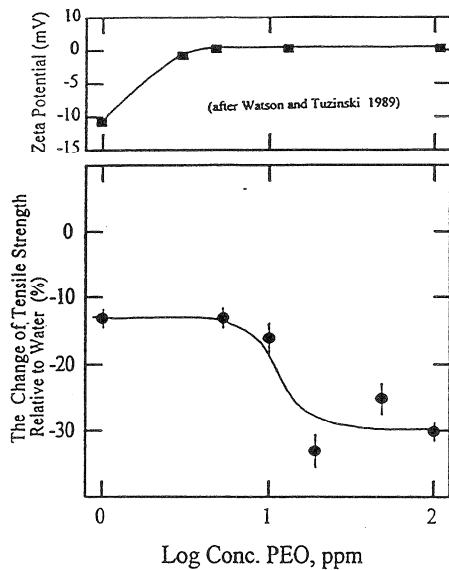


Figure 2. Variation in the change of tensile strength relative to water in aqueous PEO environments and zeta potential of Sioux Quartzite (after Watson and Tuzinski, 1989). Note that tensile strength becomes lowest in the range from 20 to 100 ppm.

Figure 3 shows the variation of tensile strength of sandstone with concentration of DTAB solution, with the graph showing the zeta potential for quartz in DTAB solutions (Macmillan et al., 1975). The tensile strength varies significantly with concentration of DTAB solution, and becomes lowest at a concentration of  $1 \times 10^{-3}$  mol/l, which is close to ZPC concentration of DTAB-quartz system. The maximum strength reduction compared to the strength of water-saturated specimen is 23 %. For  $AlCl_3$  solution, PEO solution and DTAB solution, the tensile strength of the specimens is significantly reduced when the zeta-potential approaches zero. Considering that the specimens used in the present study are very homogenous without obvious

surface cracks and pores, and that the standard deviation is less than 7 % of the strength, the tensile strength reduction is judged to be experimentally significant. Since the observed reduction in tensile strength at near the ZPC concentration compared with its strength in distilled water is so large that it may be concluded that the zeta-potential does influence the tensile strength of sandstone.

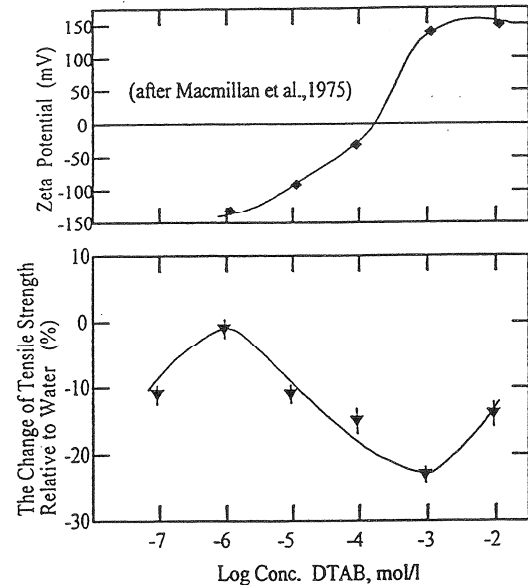


Figure 3. Variation in the change of tensile strength relative to water in aqueous dodecyl trimethyl ammonium bromide environments and zeta potential of quartz (after Macmillan et al., 1975). Note that tensile strength becomes lowest at the concentration of  $1 \times 10^{-3}$  mol/l.

Figure 4 gives a typical AE behaviour during the experiments in three different environments; dry, water, and DTAB solution ( $10^{-3}$  mol/l). The AE behaviour shown in the figure is AE event rate (counts per 5 seconds) versus time. The AE activity is the most active in dry condition, in which AE generates actively from the beginning of the loading. The least AE activity is found in DTAB solution, in which AE activity is quiet until just before fracture.

### 3.2 Triaxial Compressive Strength

The sandstone specimens saturated with  $\text{AlCl}_3$ , PEO, or DTAB solution were tested at confining pressures of 3 MPa, 5 MPa, 10 MPa, 15 MPa, and 20 MPa. Two values of chemical concentration were chosen to saturate the sample for each chemical solution; one is the critical concentration at which the tensile strength is lowest, and the other at which the tensile strength is not reduced much when compared to water-saturated specimen. In this study the multi-stage triaxial test was employed to determine the strength under the confining pressure. In this method, soon after observing peak strength relating to the first value of predetermined confining pressure, the confining pressure is increased to a desired value, and then axial stress is increased to next peak strength. The same stepwise procedure is continued until a chosen confining pressure (20 MPa) is reached.

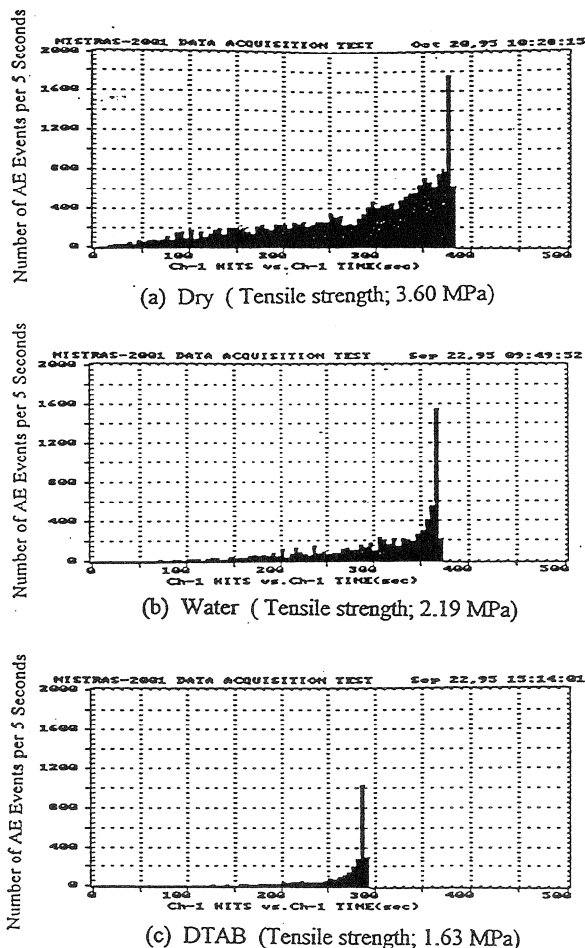


Figure 4. AE behaviours of dry sandstone, water-saturated sandstone, and sandstone saturated with DTAB ( $10^{-3}$  mol/l) during Brazilian test.

There is a statistically significant reduction in the triaxial strength in water environment when compared to dry environment. The experimental results indicate that the addition of distilled water reduce the peak strength of the sandstone by 27 %. Figure 5 represents that the reduction of the strength was quite little compared to its strength when saturated with water even at the concentration where the tensile strength is lowest. It is also found that the triaxial strength is independent of the type of chemical solution. All failures in this series of tests occurred by macroscopic shear along a single plane at an angle of approximately  $30^\circ$  to the axial stress direction. These specimens also exhibited signs of plastic deformation with significant dilation of specimen as well as the shear plane.

The AE measurement results in multi-stage triaxial tests indicate that AE activity is the most active in dry specimen, and that there is little difference between water-saturated specimen and the specimens saturated with chemical solutions.

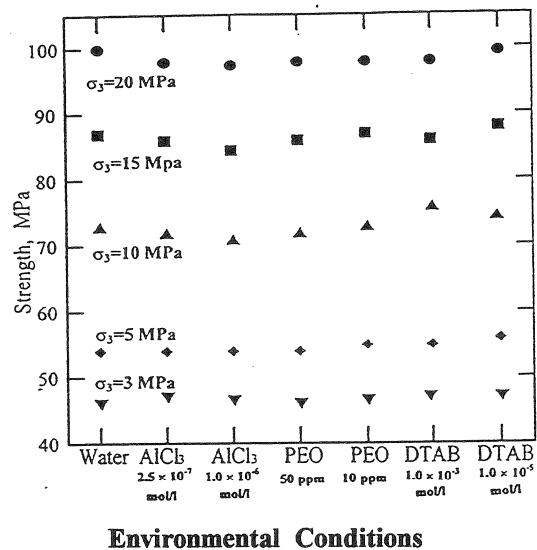


Figure 5. The strength in multi-stage triaxial compression test in water and chemical environments

### 4. DISCUSSION

The present study has demonstrated that the tensile strength of the sandstone varies significantly with concentration of chemicals, and can be markedly influenced by the zeta potential at the rock-liquid interface. The variation of the tensile strength is also consistent with the results of the laboratory scale drilling tests previously conducted by several researchers (Westwood et al., 1974; Engelmann et

al., 1987; Watson and Tuzinski, 1989). The triaxial compressive strength of sandstone, however, in three kinds of chemical environment did not vary significantly with the concentration. The reduction of the strength was quite little compared with its strength in distilled water. The possible explanation which is consistent with the experimental results is that the chemical solution affects the strength of sandstone only when the failure mechanism is dominated by tensile mode. In case of triaxial test, since shear failure mode existed predominantly during the testing, little effect of chemical solutions on the triaxial strength and no correlation between zeta potential and the strength were found in this study.

It is well known that the AE activity during the fracturing of rock is well dependent on the behaviour of rock; brittle or ductile (Scholz, 1968; Mogi, 1967; Fonseka et al., 1985). Fonseka et al. (1985) observed that in the case of the marble, which is both weaker and more ductile than silicate rocks, the number of AE events is smaller by a factor of about 50. Mogi (1967) also concluded that the brittleness of the material is the main requirement for active AE activity. Comparing the AE results in dry and saturated specimens with water or chemical solution in Brazilian test, the dry specimen represented the most active AE activity. The saturated specimen, especially the specimen saturated with chemical solution, has such low AE activity compared to dry specimen that it may be concluded that the deformation behaviour can be changed from brittle manner to more ductile. If hardness of the materials becomes the greatest when zeta-potential is zero, suggested by Westwood et al. (1974), then it is natural to expect that the deformation behaviour would become more brittle and AE activity would be more active in environments of zero zeta-potential than in environments of non-zero zeta-potential. This expectation is, however, contrary to the experimental data in this paper.

Aside from the above interpretation, the present experimental results have significant implications for mining and civil engineering. The data shows that the chemical composition of water affects only the tensile strength of the rock, and has practically no effect on the triaxial compressive strength. If the tensile fracture would be the predominant failure mode, for example in hydraulic fracturing, drilling at shallow depth, or grinding of rock particles, then fracture could be facilitated by use of a chemical solution at the appropriate concentration. The experimental results in this paper suggests, however, that the chemical components may not produce any effect on the performance in deep hole drilling.

## 5. CONCLUSIONS

Chemically induced changes of the strength of Gosford sandstone have been investigated to establish the fundamental knowledge for chemically enhanced rock fracturing. In the investigations, Brazilian tests and multi-stage triaxial tests have been performed on specimens saturated with three different kinds of chemical solution. The main conclusions are as follows:

- (1) The tensile strength of the sandstone varies significantly with concentration of chemicals, and can be markedly influenced by the zeta potential at the rock-liquid surface.
- (2) The maximum tensile strength reduction relative to the water-saturated specimen in  $AlCl_3$ , PEO, and DTAB solutions are 30.8 %, 33 %, and 23 %, respectively.
- (3) The compressive strength in multi stage triaxial tests did not vary significantly with the chemical's concentration. The reduction of the strength was quite little compared with its strength in distilled water.
- (4) The possible explanation for the experimental results is that the chemical solution produce an effect on the strength of the sandstone only when the failure mechanism is dominated by tensile mode.
- (5) The acoustic emission differs from dry specimen to the saturated specimen with water or chemical solutions. The results may suggest that the deformation behaviour changes from brittle manner to ductile.

## ACKNOWLEDGMENTS

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