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Impact of epoxy glue curing time on the quality of overcoring stress measurements in low-temperature environments

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ABSTRACT: Many techniques of stress measurement or stress monitoring are based on the principle of gluing a strain-measurement device on the wall of a pilot hole using an epoxy resin. The curing time needed for this epoxy glue to achieve full hardening is rarely put into questions. Here, we present an *in situ* study of the impact of curing time on the quality of overcoring stress measurements using CSIRO Hi cells. The tests were conducted in an argillite rock at a temperature of 12°C. We show that the conventional curing time (16 hours) is clearly insufficient in this context and leads to anomalous response in strain readings during both overcoring and biaxial tests, thus hindering stress determination. We claim that in low-temperature near-surface environments, much longer curing times may be needed in order to ensure good quality of stress measurements. Other possible strategies are discussed in the body of this paper.

1 INTRODUCTION

Knowledge of the *in situ* state of stress is of utmost importance for assessing the safety and stability of underground openings and geo-engineering structures, ranging from old dams to geological waste repositories. Yet, although much technical progress has been made over the past decades, measuring *in situ* stress in rock masses remains a challenging task (see e.g. Amadéi & Stephanson, 1997, Hakala, 2006).

One of the most widely used methods for rock stress determination is the overcoring method. It consists in measuring the strains that develop at the wall of a small diameter borehole (pilot hole) when this one is relieved from the surrounding *in situ* stress field by overcoring. Assuming a rock constitutive law (usually linear elasticity), the *in situ* stresses may be determined from the measured strains. This inversion requires the values of the rock elastic parameters which are usually determined from biaxial testing on the retrieved overcore.

Figure 1a shows the typical evolution of strains at the wall of the pilot hole during an overcoring test. The recorded strains usually show stable null readings at the start of overcoring, followed by a local maximum and/or minimum as the drilling bit passes the strain gauge position, before the curves reach plateau values which are generally used as input for stress determination. During the biaxial test (Figure 1b), strain curves normally show circumferential contraction and axial elongation as the overcore is laterally loaded. At the end of the unloading phase, the strain readings fall back to zero if the rock is perfectly elastic, or keep a slight circumferential contraction if the rock experiences permanent deformation.

Between Nov 2005 and Jan 2006, INERIS conducted an important overcoring stress measurement campaign in the argillaceous formation of the

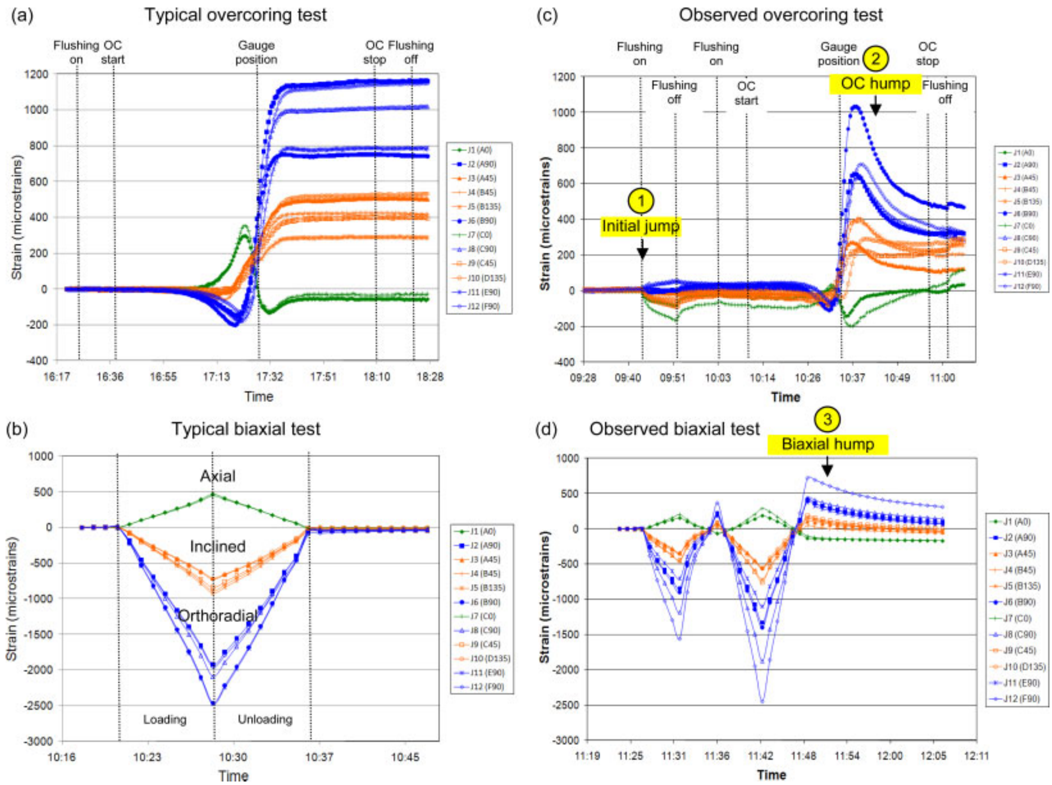

Figure 1. Left: Typical evolution curves for strains at the vicinity of a pilot hole (a) during an overcoring test and (b) during a biaxial test. Right: Strain curves recorded in Tournemire 2005–2006 experiment (c) during an overcoring test and (d) during a biaxial test.

Tournemire experimental station (Aveyron, France) using CSIRO Hi12 cells. This cell is a soft hollow inclusion of diameter 36 mm in the wall of which are embedded 12 strain gauges oriented 0°, 45°/135° and 90° from the borehole axis (see e.g. Worotnicki, 1993 for details). The cell is glued into a pilot hole of diameter 38 mm using an epoxy resin formulated according to the host rock temperature. In Tournemire, the latter is about 12°C, thus the chosen temperature range for the glue was 10–18°C. The hardening time recommended by manufacturer for this glue is 16 h.

Despite the apparent technical success of the tests and the good mechanical quality of the retrieved overcores, the strain curves recorded during overcoring and biaxial tests revealed anomalous behavior (Figures 1c, d), which practically hindered the determination of *in situ* stresses. These include (1) a systematic jump in strain readings when the air flushing system was turned on at the beginning of overcoring, (2) a pronounced sign inversion of the strain rates just after the drilling bit passed the gauge position and (3) a transient circumferential dilation of the inclusion at the end of the unloading phase.

In the following, we will refer to these phenomena as “initial jump”, “overcoring hump” and “biaxial hump”, respectively. Note that the biaxial hump was present only in biaxial tests performed immediately after overcoring (within a few hours). When the same test was conducted (on the same overcore) several days later, the biaxial hump was not observed anymore.

In this paper, we report on a new overcoring campaign carried out in the Tournemire experimental station in Nov 2008, also using CSIRO Hi cells. This experiment was purely methodological and designed to understanding the physical origin of the anomalous phenomena observed in 2005–2006. It showed that these phenomena are related to incomplete hardening of the epoxy glue at the start of overcoring.

2 DESCRIPTION OF THE EXPERIMENT

The experiment consisted in performing 6 overcoring tests in the same borehole (TC3), at a distance of 1m from each other, in a zone considered as homo-geneous in terms of rock geology (argillite) and *in situ* state of stress. The borehole was parallel to the bedding of the argillite rock, thus the CSIRO cells were oriented along the plane of mechanical isotropy of the material.

Several experimental parameters were varied along the different tests, including the overcoring speed, the rotation drilling speed, the drilling fluid (air/oil) and the curing time of epoxy resin before the start of overcoring. The only parameter which showed a significant

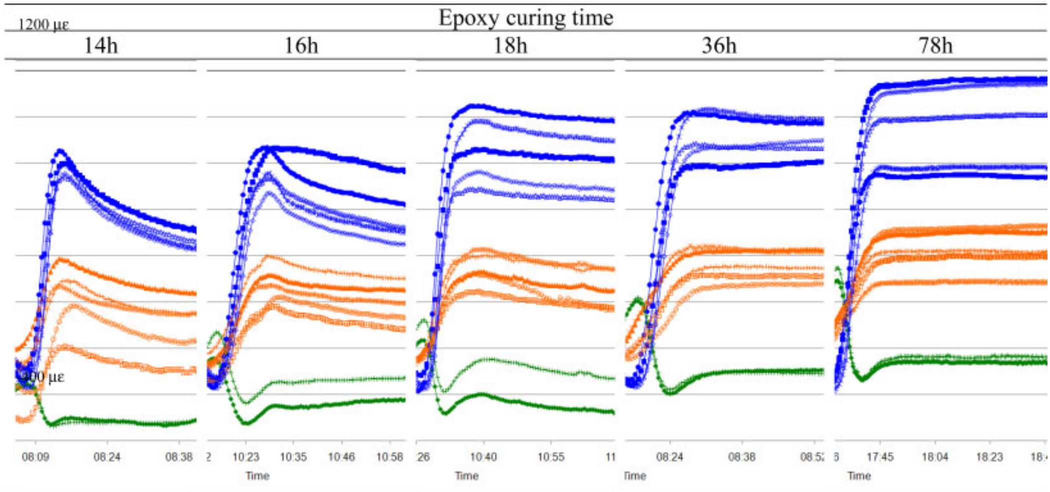

Figure 2. Curing time impact on the "overcoring hump". From left to right are shown the strain responses of CSIRO Hi cells to overcoring when the latter is performed at increasing curing times (tests TC33, TC32, TC31, TC35, TC36 respectively). Note that test TC34 failed due to technical problems. For comparison, all graphs have the same y-axis scale range. The 5 upper, 5 intermediate and 2 lower curves of each graph correspond respectively to the 5 orthoradial, 5 inclined and 2 axial gauges of the CSIRO cell.

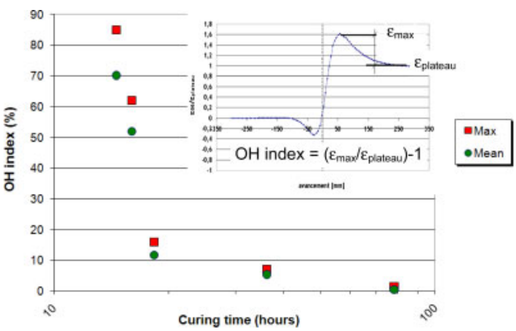

Figure 3. Curing time impact on the "overcoring hump" as quantified by the OH index.

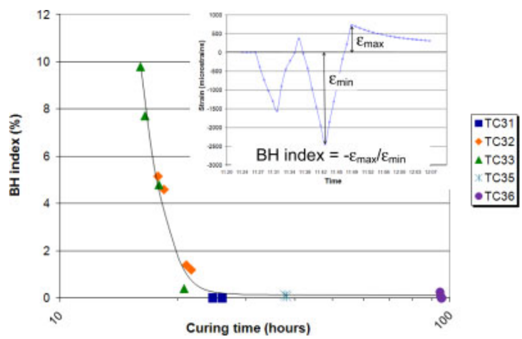

Figure 4. Curing time impact on the "biaxial hump" as quantified by the BH index.

impact on the above discussed phenomena is the curing time of the epoxy glue.

3 RESULTS

3.1 *Curing time impact on the "overcoring hump"*

Figure 2 presents the strain measurement curves recorded during all overcoring tests performed in Tournemire in 2008 (the graphs are displayed for increasing curing times). They clearly demonstrate the dependence between the curing time and the amplitude of the "overcoring hump".

Figure 3 shows the amplitude of this hump, as expressed by the OH (overcoring hump) index, as a function of curing time. We note that the hump fully disappears only after 78 h of curing.

3.2 *Curing time impact on the "biaxial hump"*

In the same way as for overcoring hump, we represent in Figure 4 the amplitude of the hump observed during all biaxial tests performed in 2008, as expressed by the BRI (biaxial hump intensity) index, as a function of curing time. The graph shows a clear dependence between the amplitude of the biaxial hump and the curing time. The curing time needed for the biaxial hump to disappear ranges between 20 and 30 h.

3.3 *Curing time impact on the "initial jump" in strain readings at the onset of flushing*

Figure 5 shows the strain measurement curves recorded at the onset of flushing for all overcoring tests performed in Tournemire in 2008 (the graphs are displayed for increasing curing times). They clearly demonstrate the dependence between the curing time

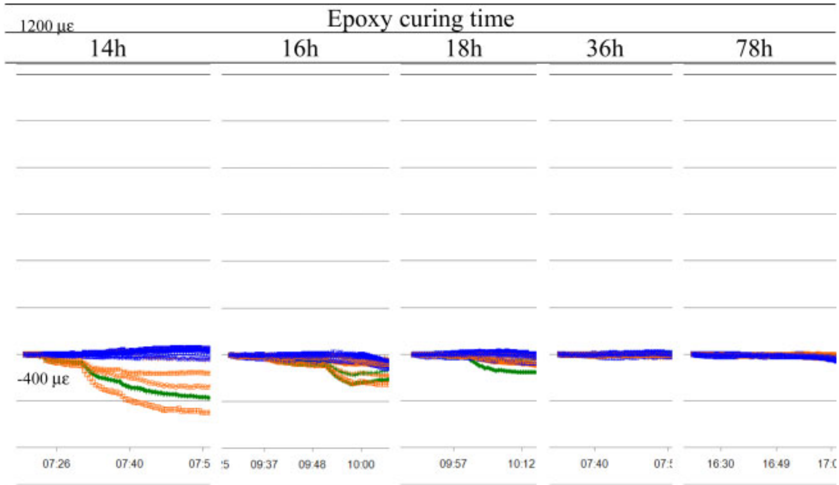

Figure 5. Curing time impact on the initial jump in strain readings at the onset of flushing. From left to right are shown the strain responses of the CSIRO Hi cells to the onset of flushing for increasing curing times (tests TC33, TC32, TC31, TC35, TC36 respectively). For comparison, all graphs have the same y-axis scale range.

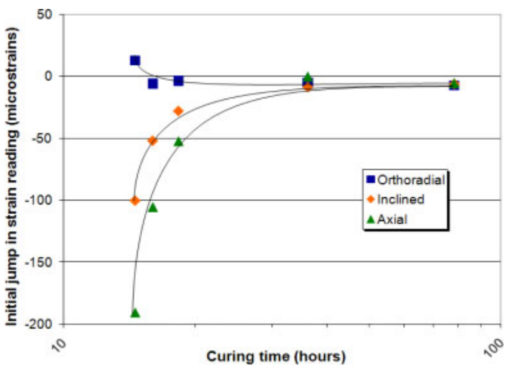

Figure 6. Curing time impact on the initial jump in strain reading at the onset of flushing. Each point corresponds to an average for all gauges of the same orientation.

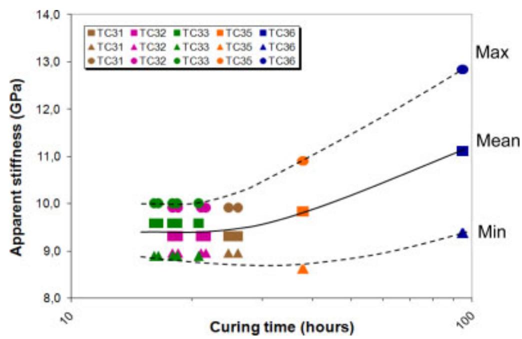

Figure 7. Curing time impact on the apparent stiffness of overcored samples determined from biaxial test.

and the amplitude of the "initial jump". As shown in Figure 6, the curing time needed for complete vanishing of the initial jump is of the order of 40 h.

3.4 *Curing time impact on the apparent stiffness of the overcore determined by biaxial test*

For each biaxial test, the apparent stiffness of the overcored sample was characterized. This was done, for each orthoradial gauge, on the basis of the secant slope of the unloading strain-pressure curve in the pressure range 0–5 MPa, by using the thick cylinder solution for isotropic linearly elastic rock cores with HI cells (Woronicki, 1993, equations 19–20). For each test, 5 stiffness coefficients were thus calculated (one per orthoradial gauge), from which the mean, maximum and minimum values were determined. Figure 7 represents those values as a function of the epoxy curing time at the start of the biaxial test. The fact that the stiffness coefficients are not the same for all orthoradial gauges comes from the transversely isotropic behavior of the rock.

We note that the apparent stiffness of the core samples change with the hardening time. This is interpreted as a signature of the change in the glue mechanical properties as it hardens. Contrary to the previous observations (overcoring hump, biaxial hump, initial jump), this change in apparent stiffness seems to hold up to very high curing times (>100 h), i.e. outside the range of curing times considered in the present study. This indicates that the hardening time needed for the glue to achieve its definite mechanical properties may be even longer than the time suggested by the disappearance of the anomalous behaviors cited above.

3.5 *Curing time impact on the average amplitude of strains during overcoring*

Beside the disappearance of the overcoring hump, Figure 2 shows that the average amplitude of peak strains

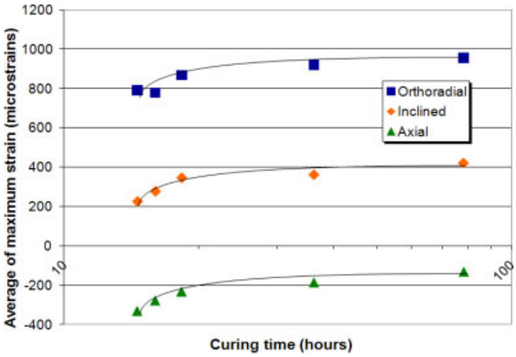

Figure 8. Curing time impact on the average amplitude of peak strains that develop during overcoring. Each point represents average value for all gauges of the same orientation.

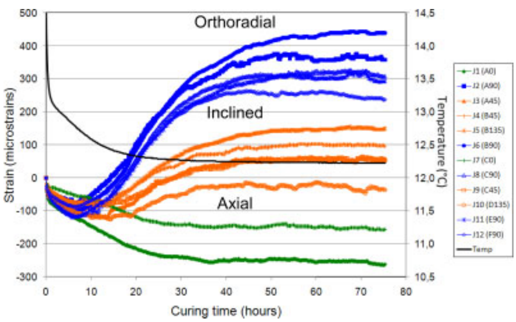

Figure 9. Typical evolution of strains and temperature recorded in Tournemire tests during glue hardening (test TC36).

that develop after the drilling bit passes the gauged area increases with the curing time. Figure 8 illustrates this in a more quantitative way, for each set of gauges of the same orientation (orthoradial, inclined, axial).

Note that this result has important implications for stress determination as it means that it is inadequate to use peak strains (for a test where the glue would not have fully hardened) instead of plateau strains, to determine *in situ* stresses.

3.6 *Strains measured during glue hardening*

To help characterize and better understand the process of glue hardening in the context of the Tournemire experiment, it is worthwhile to examine the strains recorded on the CSIRO Hi cell during hardening time (Figure 9).

The observed curves may be divided into three broad sections. In the first hours after setting (0–10 h), the temperature decreases rapidly, probably as a result of the decrease in heat production associated with glue hardening exothermic reaction. In this phase, the strain curves are essentially correlated with temperature and equal strains on all axial, tangential and inclined gauges are observed. In a second phase (∼10–60 h after setting), strain curves split according

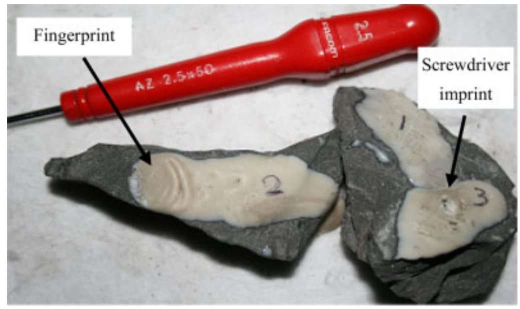

Figure 10. Glue aspect after 16 h curing (12°C) and heating under a spotlight (∼60°C) during a few seconds. The glue softens so it can be easily moved with a finger or a screwdriver.

to their gauge orientation, globally indicating lateral expansion of the inclusion. This expansion is thought to be associated with the shrinkage of the glue annulus due to hardening process, which pulls the CSIRO inclusion toward exterior. In the last phase (>60 h after setting), strain readings reach stable values.

The noticeable point here is that strains recorded during hardening time do not yield stabilization before 60 h. If we assume that strain stabilization is an indicator of the end of the glue hardening process then it may be concluded that complete hardening is not achieved in these tests before 60 h of curing.

3.7 *Direct observations on the epoxy glue*

For each test, reference gluing was prepared and let harden in the same conditions as in borehole. After 16 h, the glue was found to be relatively hard but a little sticky. When the sample was warmed under a spotlight (∼60°C) during a few seconds, the glue softened and became malleable (Figure 10).

4 DISCUSSION

Most stress measurement cells used for stress determination or for stress monitoring (Borre Probe, CSIRO Hi cell, CSIR-cell, ANZI-cell, etc.) are based on the principle of gluing the cell on the wall of a pilot hole using an epoxy resin. This solution has been experienced for decades and has proved adequate in many environments. However, in shallow-depth low-temperature environments (shallow URLs, geoengineering structures, shallow mines, natural rock slopes), the use of epoxy has been shown to be more problematic (Garrity et al, 1985, Irvin et al., 1987).

This study is a further illustration of the difficulties that may be experienced if no special care is taken to the glue hardening problem. We have shown that in a 12°C environment as the one existing in Tournemire experimental station, the use of a classical curing time (16 h) leads to anomalous behavior of strain readings during overcoring and biaxial tests (Figure 1c, d), which makes it impossible to determine *in situ* stresses.

We interpret these behaviors as being the consequence of an improper coupling between the measurement cell and the rock wall due to insufficient hardening of the epoxy glue. The fact that the glue softens and becomes malleable after being let a few second under a heat source (Figure 10) is direct evidence for the hardening process not to be completed after 16h of curing at this temperature. This is confirmed by the strain curves recorded during hardening time, which stabilize only after about 60 h of curing (Figure 9).

The empirical correlations we have built between the curing time and the amplitude of the anomalous phenomena mentioned above (see Figures 3–4, 6) enable us to establish that the hump in strain readings during overcoring, the initial jump of strains at the onset of flushing and the hump in strains readings at the end of the biaxial test, respectively disappear after a curing time of the order of 80 h, 40 h and 24 h, in the conditions of the Tournemire site. However, the evolution of the estimated stiffness of the overcored samples as a function of curing time (Figure 7) indicate that even after a curing time of 100 h (the longest curing time we have tested), the definite mechanical properties of the glue do not seem to be achieved yet. This, along with the fact that the final strain readings (so-called "plateau values") at the end of overcoring change with curing time (Figure 8), implies that undertaking *in situ* stress measurements from overcoring tests where the glue would not have fully hardened is probably doomed to failure.

To circumvent this problem, several strategies may be developed. First, longer curing times may be respected before the start of overcoring. This strategy has been adopted (with variable success) in the scandinavian URLs where the rock temperature is below 10°C and where hardening times of at least 48h (preferably 72h) were systematically respected (M. Hakala, personal communication). Second, heating of the pilot hole prior to, and during glue hardening may help significantly reduce the time needed for the glue to achieve complete hardening. This solution was recently deployed by INERIS for stress measurements in a concrete geo-engineering structure at a temperature of about 10°C. The heating system enabled the temperature to rise up to 20°C during glue hardening, leading to successful overcoring tests (no humps on strain readings during overcoring or biaxial tests, glue hardening achieved within 18 hours of curing, good quality and reproducibility of stress measurements). Nonetheless, this solution needs to be tested further in other rock materials since for certain rock types, thermal stresses due to heating may induce significant damage of the pilot hole wall. Third, glues adapted to low-temperature environments may be used (or developed). To our knowledge, this solution has not been fully explored yet. Fourth, stress measurement cells with no glue may be used. To our knowledge, three-dimensional stress measurement cells of this type do not exist. This provides a possible route of research and development for near future.

5 CONCLUSION

We have reported on a unique *in situ* study of the impact of the epoxy curing time on the quality of overcoring stress measurements. This study shows that in low temperature environments (shallow URLs, tunnels, dams, shallow mines, rock slopes), much longer curing times than the one suggested by the manufacturer may be needed in order to ensure complete hardening of the glue and therefore, good quality of stress measurements. A possible alternative strategy is to heat the pilot hole prior to, and during glue hardening. This solution was tested with success during a recent overcoring experiment in a geo-engineering concrete structure. Systematic applicability of this solution in different rock types remains to be tested.

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