

Assessment of Bond Strength of Various Cementitious Sealants for CCS Well Applications

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ABSTRACT

The bond performance of a range of cementitious sealants that are intended for use in the plugging of CO₂ storage wells (repurposed from sub-sea oilwells) is presented. To this end, a bespoke sample comprising a cementitious plug encapsulated in steel tube casing was designed to facilitate an enhanced curing of sealants at high pressure and temperature representing the conditions in a carbon capture and storage (CCS) well. The bond properties of the sealants following the enhanced curing regime were then examined using a relatively simple push-out-type bond testing protocol, with tests undertaken under elevated temperature and atmospheric pressure. In this article, a series of test results are presented, alongside the conditions of the sealant/steel-casing interface to provide an insight into the underlying mechanisms of the bond performance.

Keywords: CCS, shear bond, cementitious sealants, interface conditions.

1.0 INTRODUCTION

According to OEUK, over half of 280+ oil and gas fields in the North Sea of UK will cease their oil production by 2030 (upstream, 2023). With the increasing importance of the Net Zero agenda, there is a plan to repurpose some of these wells for the storage of CO₂, as part of Carbon Capture and Storage (CCS) operations. The use of cementitious materials is ideal in this regard due to their versatility for plugging and well-cementing in general. However, there is a concern regarding the potential of CO₂ leakage that might occur. The situation is exacerbated by the fact that the operational life for CCS well is expected to be much longer than an oil-well. **Figure 1** displays a schematic of a plugged wellbore with the potential leakage paths of liquid or dissolved carbon dioxide highlighted (Gasda *et al*, 2004). These can include (i) the external casing/sealant interface; (ii) the internal casing/sealant interface; (iii) the sealant body microstructure; (iv) fractured casing; (v) fractured sealant, and (vi) the external sealant/rock interface.

Of the interest in this work is the bond interface performance of a range of cementitious sealant materials that have the potential to be used for CCS well plugging. This article describes our ongoing attempt within the CEMENTTEGRITY project funded through ACT3 (Accelerating CCS Technologies project No 327311 – see www.cementegrity.eu). The primary aim is to investigate the performance characteristics of wellbore sealants that could ensure long term sealing effectiveness during CO₂ storage (Van Noort *et al*, 2023).

As part of this project, research is being conducted at Heriot-Watt University with the primary focus on the integrity of the sealant/steel interface (Path (i) noted above). A bespoke sample holder and bond testing protocol (Patent No. NO 20191422 and EP 3 832 290 A1)

were specifically designed to allow for the testing of a range of sealant materials that had to be cured under an enhanced curing regime that synthesizes aspects of the in-situ environmental conditions of a subsea wellbore. Example results of bond testing are presented.

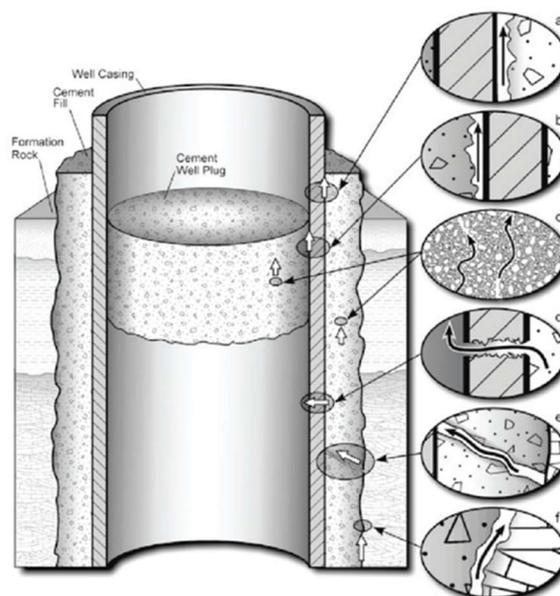


Fig.1. Schematic representation of a plugged well, showing potential leakage pathways (Gasda *et al*, 2004).

2.0 EXPERIMENTAL PROGRAMME

2.1 Materials and Sample Preparation

The sealant materials studied in this work are presented in **Table 1**. Sealant S1 is a standard Portland cement

Class G (British Standards Institution, 2000) containing silica flour (35% by weight) and is included as a baseline reference. The other sealants are proprietary materials whose details cannot be fully disclosed.

Table 1. Sealants examined in current study.

Sealant	Material Content	Description
S1	OPC with 35% silica flour	Old oilwell reference
S2	Modified S1 plus minerals addition	Currently used for low permeability application
S3	Modified S1 plus ReStone mineral	Modified blend - CO ₂ sequestering
S4	Calcium aluminate cement system	Acid resistant - used at high temperature
S5	Granite based geopolymer	1-part system designed for CCS

Sealants S2 and S3 are OPC based. However, S2 has an unspecified amount of mineral additions, designed to produce very low permeability – a desired sealant trait. S3 is a blend containing RePlug®, a mineral addition (see <https://restone.no/replug/>) conferring a CO₂ sequestering facility. S4 is a proprietary calcium aluminate system currently deployed in high temperature wells and considered highly acid resistant. S5 is a geopolymer sealant engineered specifically for CCS.

Experimental samples take the form of relatively small steel cylinders (50.8mm OD, 44mm ID, 50mm length) into which is moulded a sealant cylinder with 40mm diameter ends protruding 10mm (see **Figure 2**). The moulded shape is facilitated by two PTFE end caps which encapsulate the material during curing. Such samples are miniature versions of the sealant-plug/casing well-head represented in **Figure 1**. At the onset of the project, the use of mild steel casing instead of super duplex steel was sought by the project consortium to allow for possible occurrence of corrosion to take place, as well as to facilitate fabrication.

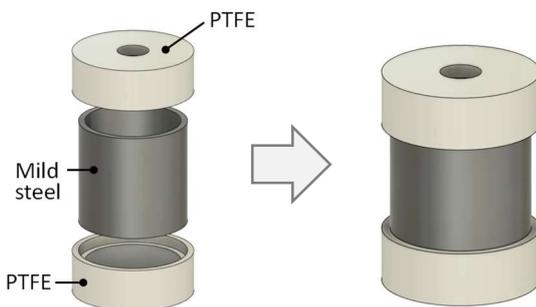


Fig. 2. Schematic diagram of sample holder.

2.2 Sealant Curing Regime

All samples were prepared and cured at the Haliburton facility in Stavanger, Norway, where autoclave equipment capable of the required enhanced curing regime is located (see **Figure 3**).



Fig. 3. Autoclaved chamber used for high temperature and high-pressure curing.

Once the material is gauged and sealed in the moulds, these are then placed in the autoclave chambers. The curing regime is summarised graphically in **Figure 4**. Initially, the autoclave system was pressurised to 300 bars. The internal temperature was then raised over a period of 4 hours to 80°C where it was held for 3 days. The temperature was then raised from 80°C to 150°C over 7 days and held at 150°C for a further 21 days. From 31.33 days, the temperature was lowered steadily from 150°C to 20°C over a period of 7 days. When the temperature reached 100°C, the internal pressure regulator was released, allowing for the chambers to reach equilibrium depending on the temperature level. The samples were removed from the autoclave after 38.33 days once they have stabilised at 20°C.

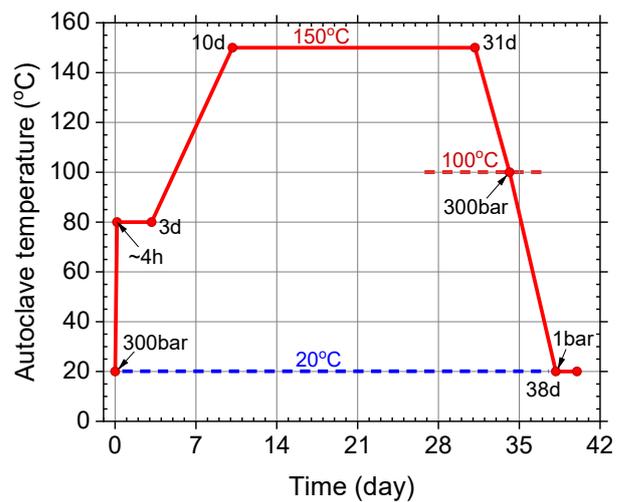


Fig. 4. Graphical summary of curing regime.

Once thoroughly cured, all samples were sent to Heriot-Watt University by air for bond testing. One day prior to testing, all sealant samples were pre-conditioned in an environmental chamber, with the temperature raised slowly to 80°C. Each sample was then immediately wrapped with an insulation, to maintain the sample temperature during testing, and tested using a 100kN universal Instron machine, following the procedure outlined in the 'Bond Strength Testing' Patent No. NO

20191422 (EP 3 832 290 A1). During testing, the load was applied on top in a load control manner, with a loading rate of 5kN/min. Testing was terminated once the plug had slid out from the steel casing.

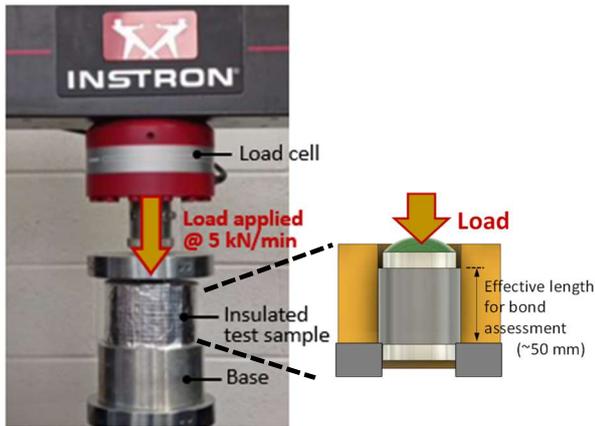


Fig. 5. Schematic of bond testing.

3.0 RESULTS AND DISCUSSION

The results from the bond testing are presented in **Figure 6** in a series of Figures, representing the results obtained from individual sealants plotted on the reference sealant (S1). In this Figure, the apparent bond strength was obtained by simply dividing the load applied on top with the contact area and plotted against the displacement measured on the top of the sample. In general terms, all sealants display a similar general increase in the Apparent Bond Strength with increasing displacement as one would expect, but the individual sealant exhibits varying strengths and stiffnesses with a general reduction in the overall stiffness and bond strength evident compared to Sealant S1. For instance, in case of Sealant S2, the average mean bond strength was 1.5MPa, which is only ~33% of that of Sealant S1 (= 4.7MPa). Sealant S3 exhibited higher average bond strength, giving an average value of 3.5MPa or 76% of that of the reference value. Sealant S4 exhibited the lowest apparent bond strength, with an average value of 0.5MPa which is only approximately 11% of that of the reference sealant. This sealant also exhibited the lowest initial stiffness, indicating possibly the occurrence of slippage during testing. The higher apparent bond strength and stiffness of Sealant S1 were however found to be associated with the corrosion that developed at the sealant/casing interface. This is discussed below.

After testing, the metal casing encasing the sealants were milled off to enable the condition of the sealant/casing interface to be observed. The results of which are presented in **Figure 7**. As can be seen, the conditions at the sealant/interface for the different sealants are remarkably different. Sealant S1 exhibited a significant development of corrosion at the interface. From a parallel investigation, it was found that this casing/sealant interface corrosion created internal pressure that artificially increased the apparent bond strength and stiffness of Sealant S1.

Sealant S2 displayed an interesting formation of white patches occurring on both the outer surface of the sealant

and the inner surface of the metal casing. The formation of these patches would suggest that the bond failure in this particular sealant must be triggered by the combination of bond failure at the interface and the failure of the sealant material itself. A trace of minimal corrosion could also be observed.

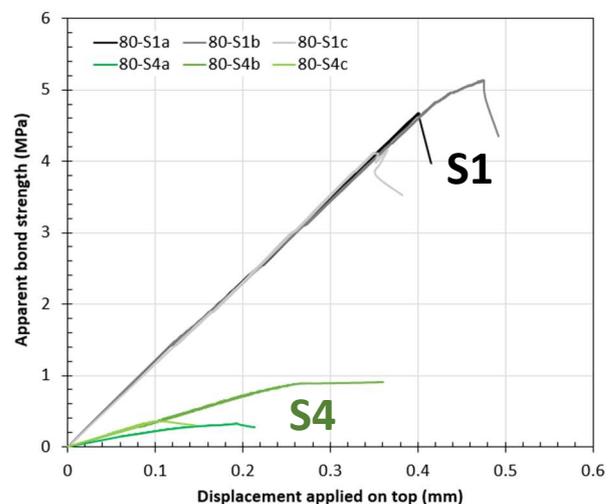
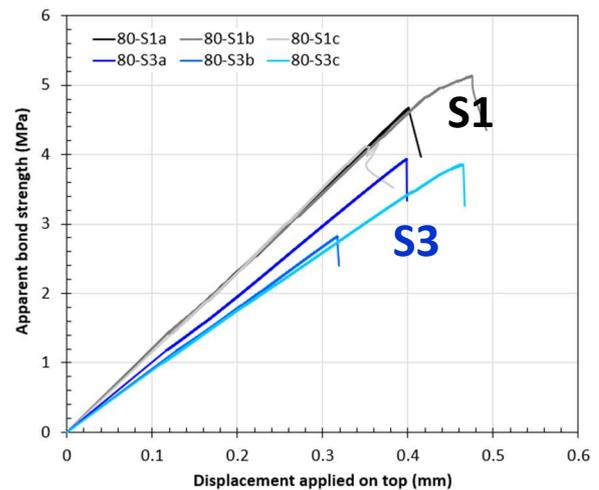
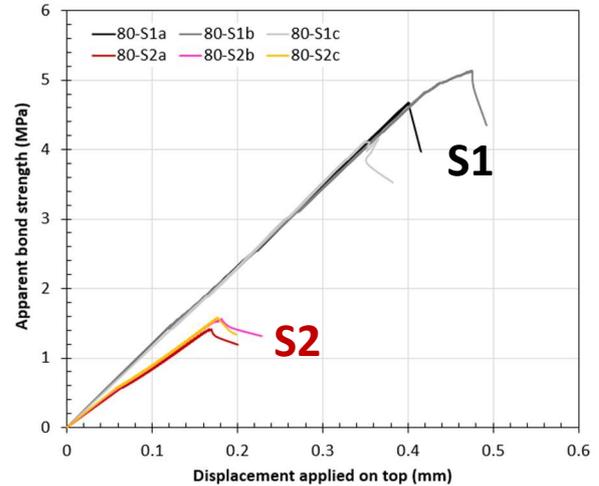


Fig. 6. Apparent bond strength vs displacement plots for various sealants.



Fig. 7. Sealant/steel interface conditions. From top to bottom: Sealants S1, S2, S3, and S4.

Sealant S3 exhibited the least corrosion amongst the four sealants tested. No formation of white patches was evident at the sealant/casing interface suggesting that the bond failure was attributed to the interface shear bond failure.

Sealant S4 displayed a notable formation of corrosion. This corrosion issue, alongside the large reduction in strength, was possibly due to the rapid setting of this particular sealant. Work is currently progressing to confirm this possible factor.

4.0 CONCLUDING REMARKS

From the investigation of four cementitious sealant materials presented in this study, wide differences in the apparent bond strength between the sealants were observed.

Sealant S1, representing material which is commonly used in well cementing, displayed the highest apparent bond strength and stiffness. It was found that the apparent bond strength and stiffness are however influenced by several different factors at the sample/steel interface, with corrosion at the casing/sealant interface appearing to be one significant factor. The elevated, apparent bond strength must therefore be treated with cautious as it might not be related to seal ability, due to corrosion contribution.

Sealant S2 exhibited a mixed mode of failure, involving bond failure at the sealant/steel interface and shear failure through the sealant material. A trace of corrosion was also evident despite relatively minimal.

Sealant S3 outperformed other sealants as it displayed relatively high apparent bond strength, with no indication of corrosion at the steel/casing interface. Failure was governed by the bond failure at the steel/casing interface. The improved performance of Sealant S3 might be related to the specific thermal properties of this particular sealant. Work is currently ongoing to confirm this possible aspect.

Sealant S4 had the lowest apparent bond strength, possibly due to the rapid initial hydration of this particular sealant (which is confirmed by the monitoring during the early curing, presented in a parallel article). This test is currently being repeated with the use of retarding agent to confirm this initial finding.

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