

Evidence for Self-restoration of Olivine Based Cement Under Geothermal conditions: Olivine Micro-aggregate as Mitigation to Portland Cement Acidic Attack

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ABSTRACT

Geothermal reservoirs provide some of the harshest conditions in which Portland cement-based materials are used. Conditions include acidic geofluids, high temperature and high pressure (AG-HT-HP). Portland cement materials were not developed for such use. However, these conditions are ideal for promoting rapid carbonation of highly mafic minerals, such as olivine. Our published studies confirmed carbonation of olivine sand under HTHP and provide potential for the long-term stability of an earth-mimicking rock material. Because of the HTHP conditions, and sufficient flow-rate of reactive CO₂ rich fluids, the mineralization in the lab occurs in a timeframe that can be over a million times faster (i.e., within hours to days) than the thousands of years such reactions require to proceed under surface conditions. To leverage this advantage of olivine, while tapping into the legacy of Portland cement for slurry delivery, rapid setting, and low matrix permeability, we explore using olivine microparticles as a micro-aggregate to Portland cement. The goal is to turn the challenging AG-HT-HP conditions as an advantage, providing acceleration of the hydration and carbonation reactions associated with olivine in order to create Portland cement with robust self-restoration ability, where olivine micro-particles react with CO₂ fluids and prevents the acid attack on Portland cement. This olivine-based cement (OBC) aims to provide more resilience of the cementing system, by disrupting chains of failure events, starting with calcium leaching and ending in compromised mechanical strength. To explore the feasibility of self-healing of OBC, a mini wellbore emulating AG-HT-HP system is constructed to conduct core-flooding tests. The monitored permeability change of OBC and Portland cement are compared, thereby demonstrating the resilience of OBC through restoration of its resistance to flow even after fracturing generates initially-conductive flow pathways.

1. Introduction

With the fast development of deep reservoir technologies in recent years, the current practice is pushing the boundaries of cementing job to more harsh and challenging locations where the acidic geofluids, high temperature and high pressure (AG-HT-HP) are prevailing. Especially in Enhanced Geothermal System (EGS), where, the high temperature and high pressure (HTHP) conditions can include wellhead shut-in pressures of more than 10,000 psi and/or temperatures higher than 300°C (Feng, Chen, & Xu, 2014); Junior, Ribeiro, and Santos (2009); (S.-M. Lu, 2018). These HTHP conditions will cause an inadequate mechanical performance of the cement barrier and the degradation of its isolation function. At the same time, the acidic geofluids (pH 2-5) that can exist in the subsurface will lead to dissolution of the Portland cement, which can cause strength reduction and physical failure (Achang & Radonjic, 2021). There exist numerous studies in the literature referring to the impact of AG-HT-HP conditions on the cement (Allahvirdizadeh, 2020; Kruszewski & Wittig, 2018; Wu, Patel, & Salehi, 2020) and it can be concluded that Portland cement materials were not originally designed and developed for use under such challenging conditions, and use of them in wells that have AG-HT-HP conditions contain environmental, economical, and technical risks.

To prevent cement failure under these challenging conditions, some recent advances (e.g. Khalifeh, Hodne, Korsnes, and Saasen (2015)) propose the use of the rock-based “geopolymer” as the cementing and barrier material. Such solutions do not use manmade polymer but instead rely on generation of binding material through geochemical processes. Binding processes include carbonation similar to that which occurs in nature. Although these products may take geological time (thousands of years) to occur, they mimic nature and, in terms of long-term stability, possess superior chemical and physical properties to man-made materials (Ashraf, 2016; De Silva, Bucea, Moorehead, & Sirivivatnanon, 2006; De Silva, Bucea, & Sirivivatnanon, 2009).

The AG-HT-HP conditions detrimental to Portland cement can be beneficial when new cements are used, such as Geologically Activated Cements (GAC) (Y. Lu, Spencer Williams, Frizzell, & Bunger, 2020). This approach is inspired by the work of Kelemen and Matter (2008) who studied the reaction kinetics of the carbonation reaction of the ultramafic minerals. They concluded that under certain ranges of temperature and pressure, the geochemical alteration rate of the ultramafic minerals can be increased by a factor of 10^6 . Hence, a process that takes tens of thousands of years can be accelerated to hours or days, provided chemical environment and the temperature-pressure conditions are suitable. Fortunately, these ideal temperature-pressure ranges happen to be similar to the HTHP reservoir conditions in many EGS wells (and deep-water oil wells). The experimental work of Lu et al. (2020) has demonstrated the feasibility of GAC’s genesis and self-healing by placing the pulverized ultramafic mineral into a small-scale, wellbore-emulating batch reactor allowing them to hydrate and carbonate under HTHP conditions. Their result demonstrates that combining CO_2 and Mg_2SiO_4 at HTHP conditions mimics the natural process of olivine weathering via dissolution and subsequent carbonate precipitation, but on a 10^6 more rapid time scale. More importantly, they show that in the presence of corrosive fluid, the GAC has the self-healing ability under conditions of HTHP, which is evidenced by the decreasing flow rate through a damaged sample over time. Furthermore, the recent work of Achang and Radonjic (2021) discovers that olivine reacts with low pH solution between 1.0 to 1.92 and has no significant reactivity in other fluids with

different pH, i.e., deionized water (pH 7.58), sodium chloride (pH 9.30), calcium hydroxide (pH 12.60), synthetic oil (pH 5.00) and it is also not reactive with cement.

This work aims at exploring the feasibility of creating the new resilient Olivine Based Cement (OBC) by using olivine micro particles as a micro-aggregate in Portland cement. The goal is to leverage the challenging AG-HT-HP conditions as an advantage, providing acceleration of the hydration and carbonation reactions associated with olivine in order to create Portland cement with robust self-restoration ability, where olivine micro particles react with CO₂ fluids and presents the acid attack on Portland cement. This OBC aims to provide more resilience of the cementing system, by disrupting chains of failure events that is otherwise occur on CO₂ attack. At the same time, high temperature (90°C) triaxial and creep tests are also performed on OBC cylindrical samples. From these experiments, mechanical and hydraulic properties are obtained and then compared with neat Class-H cement that are cured at the same conditions.

2. Experimental Methods

Class H-Portland cement was used for all the cement slurries and was prepared as per the American Petroleum Institute (API, 2013). Neat cement slurry at a density of 16.4 ppg (1.94 g/cm³) and the water-cement ratio of 0.384. Next, 5% bwoc (by weight of cement) olivine cement slurry was prepared with a water to cement ratio of 0.404. Dispersant CFR-3 without defoamer and D-Air 5000 were added to both slurries. The slurry was placed in a mixer (Waring commercial CB15 N) for 15 seconds at a mixing speed of 1200 rpm, after which the speed was increased to 4500 rpm for 45 seconds. Samples were then poured into molds and hydrated for 24 hours. They were then de-molded and placed in Ca(OH)₂ (pH ~13) and cured in a ESPEC environmental chamber (model ENGL12-4NA1) for 21 days to gain strength in Ca(OH)₂ at 90°C and 95% relative humidity. Then all the samples were kept in pH 13 solution at ambient conditions until the time of testing.

A cement sample was mounted on the Multiprep™ system and aligned properly for accurate grinding. The Allied HighTech multi prep polisher and fluid dispenser were used to realize polishing starting with a 600 grit silicon carbide (SiC) abrasive disc used for grinding to remove initial deformations. After each step, the surfaces were inspected under the microscope to ensure a uniform scratch pattern. Grinding induced deformation was removed using 6 μm diamond suspension on Gold Label polishing cloth and 1 μm diamond suspension on White Label polishing cloth, with PurpleLub™. For water-sensitive samples a low viscosity, ethyl alcohol-based polishing lubricant (PurpleLube™) was used. For final polishing, 0.05 μm water-free colloidal silica suspension was used on a Chem-Pol polishing cloth. Additionally, samples were sonicated with isopropyl alcohol on a tabletop sonicating bath for 5 min at the end of each step to remove fragmented cement, residual diamond suspension, and colloidal silica. The polished samples were then dried overnight in a drying oven at 50°C before carbon coating for scanning electron microscope (SEM) analysis. Cement plugs were also fractured and a fresh surface was collected and coated with carbon for SEM analysis. All polished and fractured samples were imaged with the FEI Quanta 600 field-emission gun Environmental Scanning Electron Microscope in both secondary electron mode and in the backscattered electron mode. Elemental spots, maps, and spectra mode analysis were obtained using the Bruker energy dispersive spectroscopy (EDS) X-ray microanalysis system at 20 KeV.

Triaxial tests were performed in a temperature-controlled Hoek-type triaxial compression cell, which consists of three main parts: axial loading system, confining stress system, and temperature system (Figure 1). The experimental approach entailed applying confining pressure and temperature replicating downhole conditions while simultaneously imposing the increasing axial (deviatoric) load until the specimen failed. The deviatoric loading was controlled by an INSTRON-600DX load frame, which can provide up to 600KN. The confining stress was maintained by a high-pressure syringe pump (ISCO-260D), which was also allowing precise measurement of the volume change of the specimen associated with a given confining stress up to 70 MPa. The temperature was provided by wrapping the Hoek cell with the heating tape that can provide a controlled temperature up to 180°C. In the experimental procedure for performing the triaxial tests, the cylindrical specimen was kept in fully saturated conditions until the test starts. Then the core specimen was placed into the membrane/core holder, which was attached to the cell so that the whole system remains airtight. After the core holder with a specimen was placed into the Hoek cell, hydraulic oil was pumped into the space between membrane and cell so the lateral pressure can be provided. The specimen was axially enclosed by two steel loading plates (top-loading plate and bottom spacer). The top vertical piston of the load frame directly contacted the top-loading plate so the deviatoric load can be applied to the specimen.

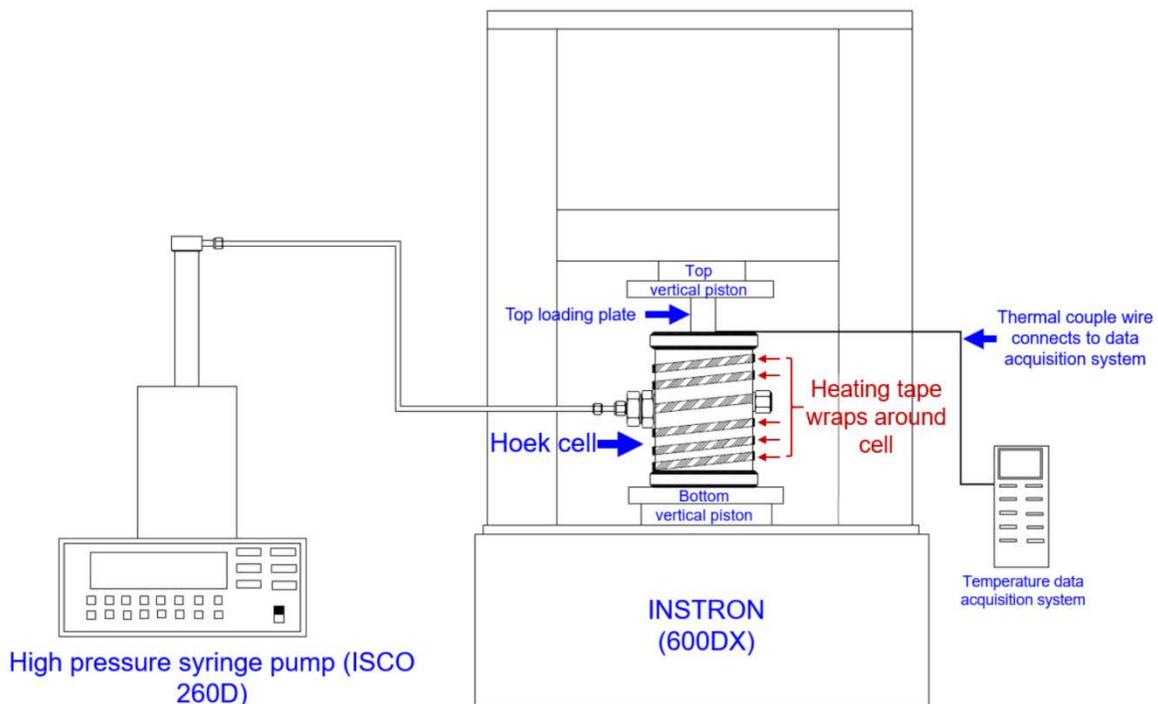


Figure 1. High-temperature triaxial test apparatus

By using heating tape wrapped around the Hoek cell, the temperature of the cell was slowly increased to the targeted temperature (90°C) over two to three hours at a low hydrostatic pressure of 1.4 MPa. After the desired system temperature was achieved and stabilized, the confining pressure and vertical load were increased to the targeted downhole pressure value, so the specimen was initially loaded isotropically, and then the deviatoric load was increased until the specimen fails. Following ASTM-D7012, the specimen was tested at a constant rate (3.3×10^{-6} m/sec) so that specimen failed in approximately 10 to 15 minutes. During the test, data acquisition recorded the load and the axial position of the top piston. These data were used to derive the axial stress and axial strain.

The creep tests were performed under the same setup with the triaxial test except replacing the INSTRON-600DX with an MTS-810, which has better long-term stress and displacement control. Following the protocol that created by Rassouli and Zoback (2018), a multi-stage creep tests over different time windows was adopted to extract different components of the total deformation, i.e. elastic, plastic, visco-elastic, and visco-plastic. The time-cycling creep tests consisted of two individual cycles and each cycle includes loading, creep, unloading and recovery phases. The sample was first subject to a constant hydrostatic pressure of 13.7 MPa and holding for at least 24 hours to reach the thermal equilibrium inside the system. The first cycle started with applying the $P_{\text{deviatoric}}$ of 13.7 MPa, ramping up over 120 seconds and then holding for 7 hours before releasing and allowing for recovery. The second cycle was the same as the first except holding the $P_{\text{deviatoric}}$ for longer time to determine the visco-elastic and visco-plastic deformation. All the samples were tested at a constant confining pressure of 13.7 MPa and a constant deviatoric pressure of 13.7 MPa for better deformation comparisons among different samples.

The self-healing experiment entailed first inducing damage to the OBC by inducing cracks. The cracks that were generated during the triaxial test are found to be the most convenient because such a work flow did not require removal of the OBC from the membrane in which the cracks are generated. This avoided displacing crack surfaces through specimen handling and reloading. After inducing cracking, the self-healing experiments were performed with a setup that is very similar to the triaxial test. The differences were the Hoek cell was placed in the oven which provided the temperature control while both ends of the Hoek cell were pressurized by two syringe pumps. The instrumentation on these pumps enabled monitoring and recording of the pressure change and the flow rate in the system. The penetrating fluid that was used in self-healing test was analogous to CO_2 –rich formation fluids, where the CO_2 is dissolved into distilled water at the pressure of 1.03 MPa to form the carbonic acid. This pH of this penetrating fluid was around 3 and the starting flow rate was controlled around 0.02-0.05 ml/min at the beginning of the self-healing test.

Before the self-healing test was started, the upstream pressure and downstream pressure were both increased to 13.7 MPa and the oven was set to 180 °C followed by a brief (60-90 minutes) waiting period for the temperature of the specimen to equilibrate to the oven temperature. After that, the pressure at the upstream was increased by a certain amount thus this pressure difference will drive the fluid flow from upstream, penetrating the cracked specimen and flowing to the downstream side of the specimen. Immediately after the upstream pressure was increased, the flow rate was monitored and measured using the downstream syringe pump displacement. The testing time for the self-healing of OBC usually took up to 10 hours and the pressure at upstream

and downstream and the temperature remained unchanged during testing, controlled independently by two syringe pumps.

3. Experimental Results

Cement hydration products and the accompanying microstructure is traditionally examined using SEM, which can be performed on either fractured or polished surfaces. To evaluate chemical integrity of olivine particles within highly alkaline Portland cement matrix, we compared polished sample surface of neat to 5% Olivine-Cement sample, as shown in Figure 2. The BSE micrograph obtained from a neat cement sample shows bright unhydrated cement grains, outlined with blue square shaped boxes, while hydration products (labelled by yellow triangle) are pointed with yellow triangles. Black diamonds (labelled by red circle), on the other hand, depict microporosity. The BSE micrograph obtained from polished cement samples containing 5% Olivine, has one additional component outlined with red circles. These indicate crushed olivine mineral particles. The images show that olivine particles are comparable to cement clinker particles on average 20-30 μm in size. Based on these images there is no observable evidence of olivine reactivity with cement.

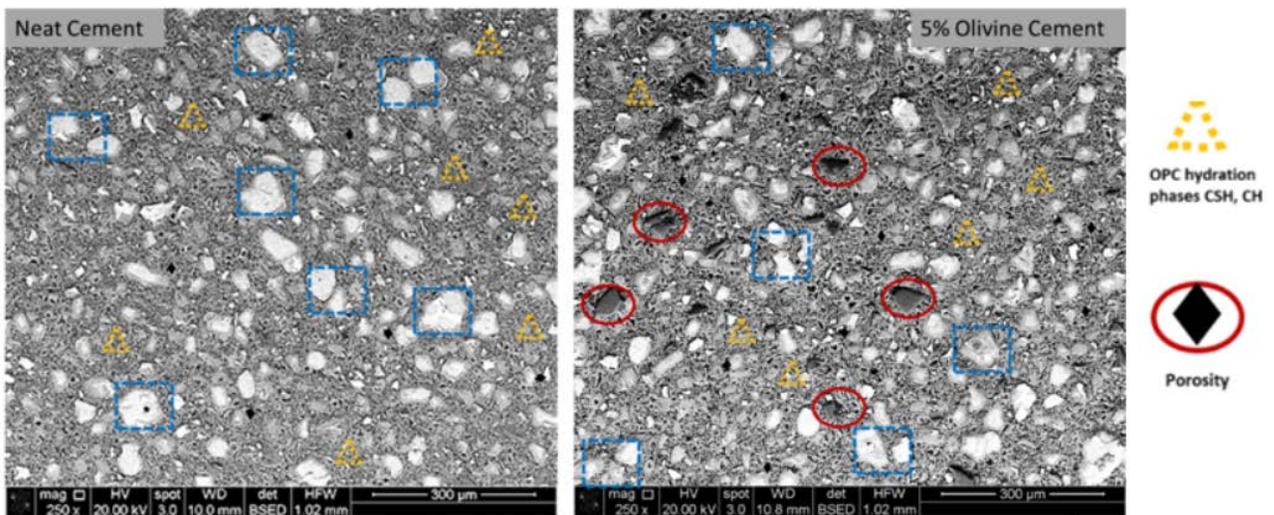


Figure 2. Back-scattered electron micrographs of neat (left) and 5% olivine cement (right) obtained from polished sample surfaces, on wellbore cement slurries made at 16 ppg and cured for 21 days at 90 °C and 95% relative humidity.

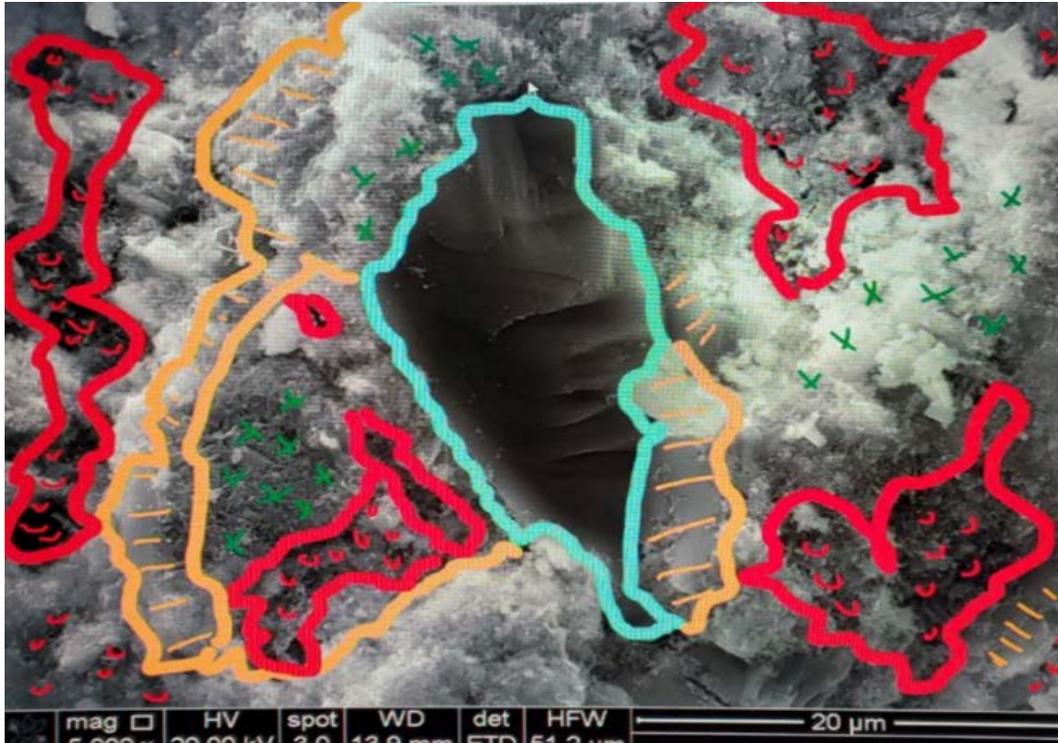


Figure 3. SEM image shows that Olivine particles do not have negative impact on cement hydration for cement slurry design and curing conditions.

At the same time, SEM micrographs and EDS profile lines are used to observe the microstructures of the olivine based cement and identify the chemical components. As shown in Figure 3, it is observed that olivine particles do not have negative impact on cement hydration for cement slurry design and curing conditions we used. Rather, olivine particles are surrounded by two density regions of CSH and well packed micro-architecture within vicinity of olivine, where no extra porosity, fractures or reduced amounts of hydration products are formed. This observation is also supported by other experimental results we obtained in present work.

The raw data of the triaxial test in this study include the dimension of the sample (sample height, H ; sample diameter, D), the lateral pressure (σ_3), the axial load (P), the axial deformation (ΔL), and the duration of the test (T). The axial stress is derived by dividing the axial load with the specimen's cross-sectional area. Additionally, using the axial deformation data from the load frame, the specimen's axial strain (e_A) can be estimated by using axial deformation divided by sample height.

Thus, the strain-stress curve of each specimen can be obtained, and are shown in Figure 4. Note that in all cases, H is taken as the initial length taken at the ambient stress conditions. To facilitate direct and clear comparison, the axial strain is taken as zero when the deviatoric stress starts to increase from zero. The Young's modulus of each sample is obtained from the linear portion of the stress-strain relationship.

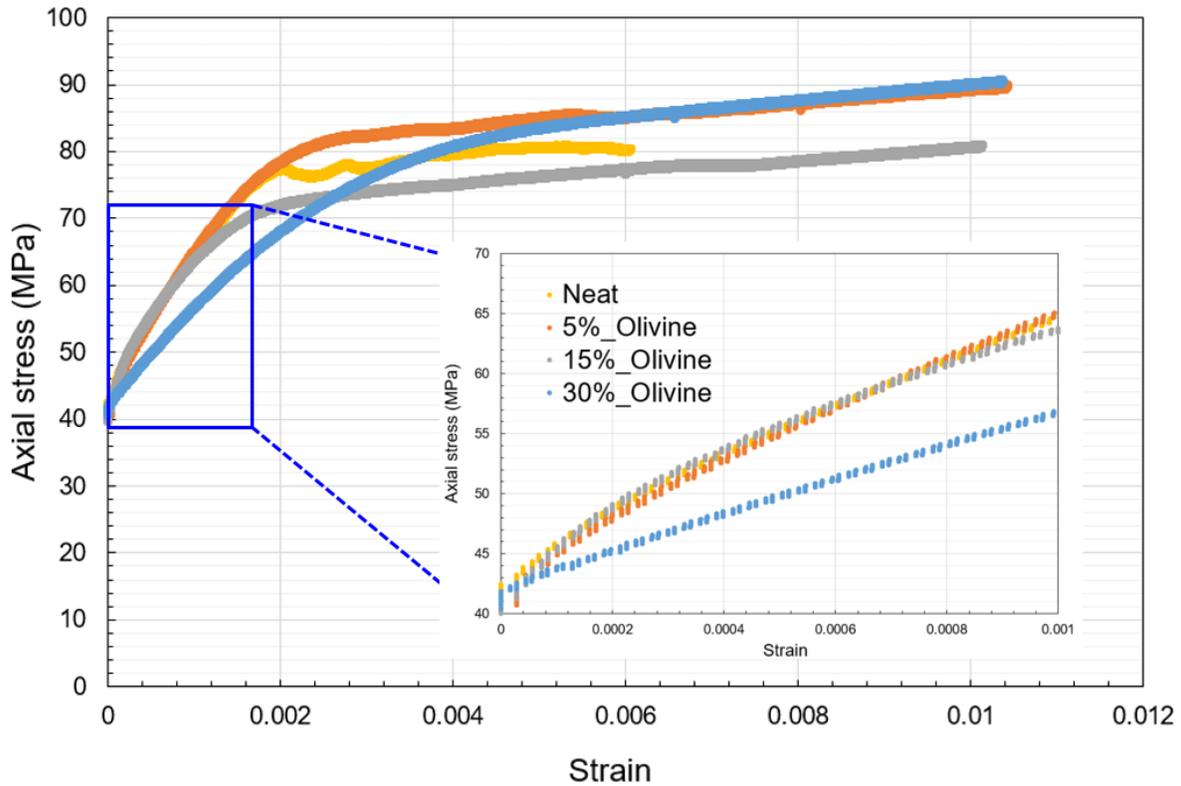


Figure 4. Strain-stress curve of neat cement, 5% OBC, 15% OBC and 30% OBC tested at 41.3 MPa and 90°C. The initial linear regime is shown in a magnified scale in the inset and the slope of this initial linear regime is used to calculate the Young's modulus.

By using the multiple stage creep tests with different time windows, the time-strain curve of neat cement and 5% OBC is plotted in Figure 5, from which the elastic, plastic, creep strain component can be determined. Although it is well-known that creep is a long term behavior, the large amount of creep deformation occurs at the early stage (first days) after applying the deviatoric loading (Acker, 2004; Rassouli & Zoback, 2018; Wyrzykowski, Scrivener, & Lura, 2019). It was observed in present experiments that, in the timeframe of two days, the neat cement has larger creep deformation than the 5% OBC when they were tested at 90 °C. The neat cement's creep deformation goes up to 69% of the total deformation whereas the OBC barely has creep deformation.

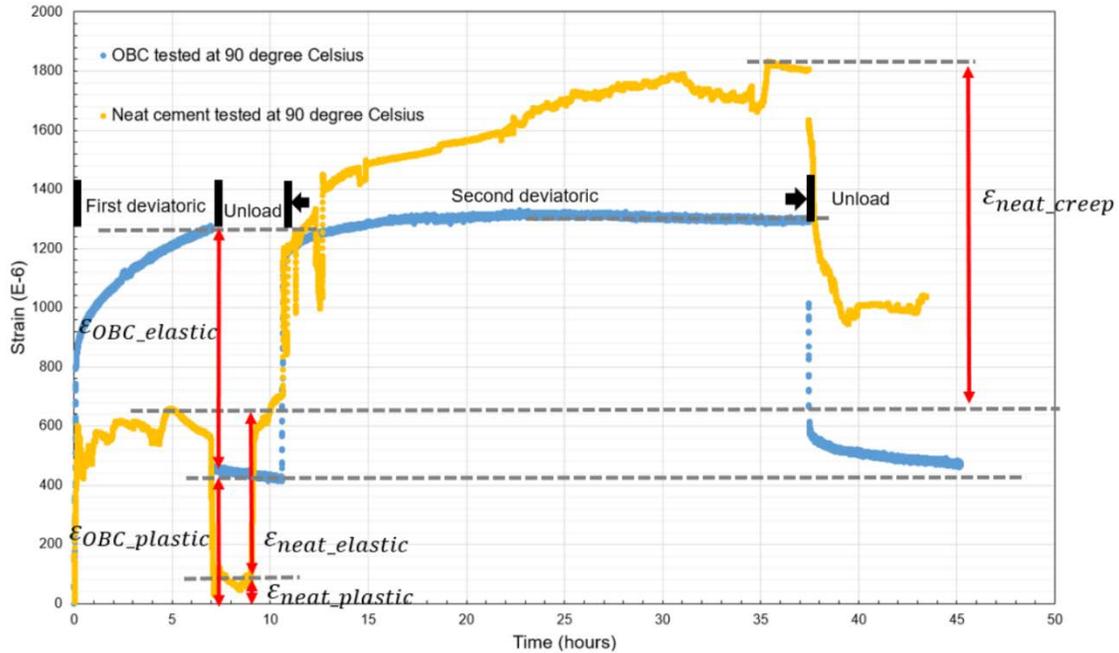


Figure 5. Example of creep data of neat cement tested at room temperature and 90°C and 5% olivine based cement (OBC) tested at room temperature and 90°C.

The permeability data of neat cement and 30% OBC are recorded in Figure 6 (a) and (b), respectively. Both of the OBC and neat cement are showing the permeability reduction after several hours of self-healing tests. However, the permeability reduction percentage of OBC is around 45%-50%, which is larger than the 15% permeability reduction percentage of the neat cement.

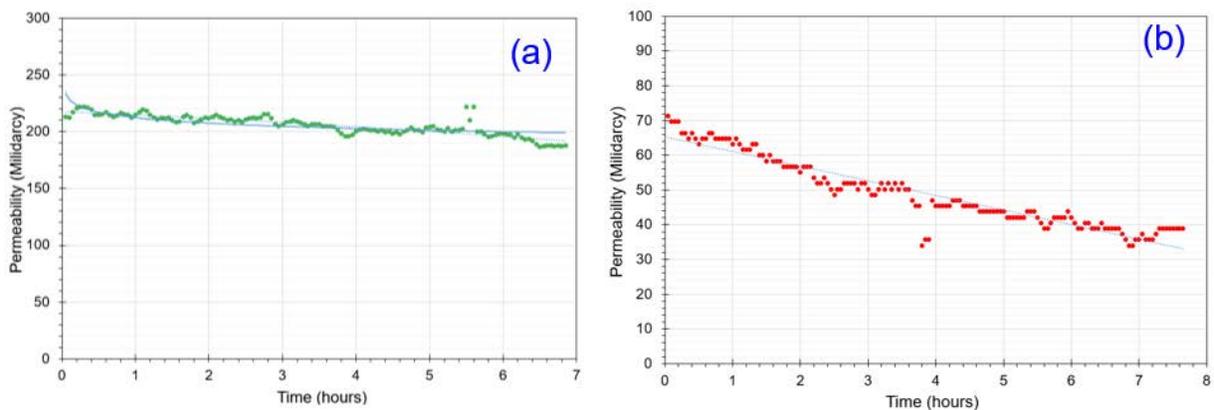


Figure 6. Self-healing data of neat cement and 30% OBC tested 13.7 MPa and 180 °C. (a) Permeability change of neat cement; (b) Permeability change of 30% OBC.

4. Discussions and Conclusions

A series of experiments including triaxial tests, creep tests and self-healing tests of OBC and neat cement were performed at reservoir like HTHP conditions. Various mechanical and hydraulic properties of different percentages of OBC were obtained. Based on the experimental results from the high temperature triaxial tests, under the same testing conditions of 41.3 MPa and 90°C, the Young's modulus of 5% OBC and 15% OBC have a 0.5% - 1% difference when comparing with the Young's modulus of cement; these differences are essentially negligible. However, when the olivine micro particle percentage is increased to 30%, OBC becomes more compliant, evidenced by the Young's modulus reducing by 12.4% compared with neat cement. Thus, it is observed that the OBC will become more compliant if the olivine micro particles which is added into the neat cement exceeds a certain amount, i.e. 30%, according to the results of current work. However, a detailed olivine dose studies are required to clarify this issue.

The creep experiments presented here show that the OBC has significantly different long-term behavior than the neat cement under HTHP conditions and these behaviors are considered as beneficial factors that will contribute to the performance of a geothermal cementing system. From strain evolution data (recall Figure 5), it is observed that neat cement will have more creep deformation compared with OBC when they are subjected to the same loading and temperature conditions. Namely, at 90°C, the neat cement's creep deformation goes up to 69% of the total deformation whereas the OBC barely has creep deformation. It is concluded that neat cement appears to have higher susceptibility to creep than the OBC, although confirming this behavior at other temperatures remains for future work. The underlying creep mechanisms of neat cement and OBC presented in this paper also remain unknown and are topics for future work. However, if neat cement has substantially larger amount of creep deformation at high temperature, as evidenced here, it may lead to the micro-fractures that initiate and develop inside the cement thereby jeopardizing the integrity of the cement system (Denarié, Cécot, & Huet, 2006; Schlappal, Schweigler, Gmainer, Peyerl, & Pichler, 2017; Waanders, Janssen, Mann, & Verdonschot, 2010). At the same time, some researchers proposed to form an effective well barrier by using the creep properties of either cement or shale (Achang, Yanyao, & Radonjic, 2020; Vrålstad et al., 2019). A caveat, though, is that on the creep data for shale (Benge et al., 2021) indicates the creep deformation of neat cement will be 3-10 times larger than the shale at reservoir-relevant temperatures. These time-dependent deformation difference will more than likely occur at the interface of cement and shale, which will put the already vulnerable place even more at-risk.

It is observed that both neat cement and OBC have the permeability reduction in the self-healing experiments. However, our results shows that the reduction percentage of OBC is higher than the neat cement. Based on the large creep deformation that is observed in high temperature creep test, the permeability reduction of the neat cement is likely caused by the creep deformation that will close the fractures and decrease the flow rate through a damaged sample. Thus, the neat cement which has more potential to creep should have the higher permeability reduction. However, the OBC, which is less susceptible to creep, has greater permeability reduction than the neat cement. It indicates that the OBC can effectively seal the breakage flow paths by other means, most likely with the products of the mineral carbonation reactions.

In summary, these results show that olivine can serve as a micro-filler in cement and be available to react with CO₂ in presence of a low pH carbonated brines to form carbonates thus maintaining

cement strength and preventing leaching of Ca-rich cement hydration products, enhancing wellbore cement resistance to leakage. The result is a self-healing material. Additionally, the OBC is less prone to creep and, at high percentages, more compliant than neat cement. Our results thus show that OBC is promising to provide improved performance under challenging conditions relevant to geothermal wells (high-pressure, high-temperature, and acidic geofluids). It also demonstrates that the OBC has potential to be resilient to failure due to self-healing, and therefore it is promising as an advanced material for the next generation of resilient well bore cementing and plugging systems.

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