Geomechanics and Engineering, *Vol. 15, No. 1 (2018) 615-620*
DOI: https://doi.org/10.12989/gae.2018.15.1.615

Self-healing capacity of damaged rock salt with different initial damage

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(Received October 13, 2016, Revised August 21, 2017, Accepted November 8, 2017)

Abstract. In order to analyze the healing effectiveness of rock salt cracks affected by the applied stresses and time, we used the ultrasonic technology to monitor the ultrasonic pulse velocity (UPV) variations for different initial stress-damaged rock salts during self-healing experiments. The self-healing experiments were to create different conditions to improve the microcracks closure or recrystallized, which the self-healing effect of damaged salt specimens were analyzed during the recovery period about 30 days. We found that: The ultrasonic pulse velocity of the damaged rock salts increases rapidly during the first 9 days recovery, and the values gradually increase to reach constant values after 30 days. The damaged value and the healed value were identified based on the variation of the wave velocity. The damaged values of the specimens that are subject to higher initial damage stress are still keeping in large after 30 days recovery under the same recovery condition It is interesting that the damage and the healing were not in the linear relationship, and there also existed a damage threshold for salt cracks healing ability. When the damage degree is less than the threshold, the self-healing ratio of rock salt is increased with the increase in damage degree. However, while the damage degree exceeds the threshold, the self-healing ratio is decreased with the increase in damage.

Keywords: rock salt; self-healing; damage; ultrasonic wave

1. Introduction

Underground salt caverns in rock salt have been provided for the storage of liquid and gaseous hydrocarbons and chemical stocks for many years (Lux and Eberth 2007, Lüdeling 2015, Liu *et al.* 2016). A good way to reserve gas in salt caverns is enhanced by (1) encapsulation of the gas by room closure due to salt low permeability, and (2) an effective sealing system in the shafts that connect to the underground rooms. Excavation of salt caverns and the process of gas injection and production, however, can induce the formation of microcracks under low confinement conditions (Houben *et al.* 2013, Wang *et al.* 2014, Chen *et al.* 2016a, Chen *et al.* 2016b, Fan *et al.* 2016). Preexisting microcracks introduced in salt during excavation represent the major channels of gas escape. The presence of damage with different type in salt can alter the structural stability and the permeability of salt, affecting the integrity of salt caverns. Thus, the development and healing of damage in salt are important factors affecting gas sealing. And it is of great importance to understand the self-healing property of rock salt damage and the influence of damage on the self-healing and what kind of damage are harmful to the mechanical properties and permeability of the cavern.

Breugel (2007) and Wu (2012) summarized the researches of self-healing in cementitious materials: self-healing or autogenous healing of cracks in fractured concrete has been noticed by the French Academy of Science in 1836 already in water retaining structures, culverts and pipes. The self-healing was also investigated. (Hearn 1998, Voyiadjis *et al.* 2011, Yildirim *et al.* 2015) and a distinction was made between self-healing and self-sealing at that time. In the first case the original strength of the concrete is completely recovered, whereas in the second leaking cracks are just closed but no strength recovery is obtained (Zhong and Yao 2008, Tittelboom *et al.* 2012).

Some researchers emphasized the dynamic competition between grain growth and grain size reduction or grain nucleation processes, and these models contain activation energy terms that show temperature dependence (Desbois *et al.* 2010, Desbois *et al.* 2012a). Damaged rock salt, which generally manifests in the form of microcracks, can be recovered or healed when subjected to sufficiently high pressures and temperatures (Chan *et al.* 1998, Ter Heege *et al.* 2005). It was found that rock salt creep damage is usually characterized by microcracks that can be self-healed by recrystallization under suitable temperature and pressure. The constitutive model coupling creep, damage, and healing in rock salt was formulated by considering individual mechanisms that include dislocation creep, shear damage,

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http://www.techno-press.org/?journal=gae&subpage=7 ISSN: 2005-307X (Print), 2092-6219 (Online)

tensile damage, and damage healing (Chan *et al.* 1996, Chan *et al.* 2001).

From the microscopic point of view, the self-healing process of damaged rock salt is the recrystallization of rock salt crystals (Peach *et al.* 2001, Ter Heege *et al.* 2004, Ter Heege *et al.* 2005).The basic processes involved in recrystallization are the migration of existing grain boundaries and the formation of new high-angle grain boundaries (Drury and Urai 1990). Grain boundary migration and subgrain rotation can occur in recrystallization. The relative importance of migration is dependent on the deformation conditions (i.e., temperature, strain rate, and stress) (Chen *et al.* 2013). Solution-precipitation creep is important for the recrystallization of creep damage in high-moisture capacity rock salt. The mechanisms is related to the various ways in which two basic processes, namely, grain boundary migration and new grain boundary formation, are combined to transform the microstructure (Urai *et al.* 1986). Some stable functional relations between average grain boundary size and yield strength were established through a large number of studies. On the other hand, the recovery condition like water content, temperature and strain were also influential to the healing effect (Fuenkajorn and Phueakphum 2011, Koelemeijer *et al.* 2012). Rock salt with low water content continuously hardens with increasing strain and is stronger than wet rock salt. The recrystallized grains at higher strain are generally coarser and more idiomorphic than at lower strain.

Relevant studies (Hou 2003, Lux 2009, Zhu and Arson 2014) showed that, although the strength and permeability of damaged rock salt could be regained to a large extent, the ability of strength recovery was determined by many factors, such as the composition of rock salt, the damaged age, damage degree, conditions of self-healing (humidity, temperature) and healing time, among which the damage degree is one of the most important factors. Thus it is very important to analyze the recovery effect of damaged rock salt with macroscopic and quantitative method.

The purpose of this study was to assess the effects of time, and initial damage level on self-healing process. Microcracks of salt specimens were created during uniaxial compression, and the wave velocity variation for these damaged salt specimens with different initial damage during the self-healing experiment was recorded. The wave characteristics were analyzed on different levels of initial damage and different healing effect of recovery stage, and the relationship between damage degree and self-healing ratio of rock salt was established.

2. Experiments

2.1. Specimen preparation

This experiment was designed to determine the influence of damage level on crack closure and healing in salt caverns. The salt specimens are high-purity salt about 96.3% to 99.8% soluble content (mainly NaCl) from the Khewra salt mine in Pakistan. These salt specimens were pink, transparent, fine grain, and compact. All specimens were processed into cuboid with 50 mm long edges (Fig. 1(a)).

(a) salt specimens (b) UPV-1 ultrasonic detector

Fig. 1 The specimens and test device

Ultrasonic pulse velocity and attenuation have been used extensively as indicators of crack development in geologic materials. The ultrasonic device UPV-1 (Fig. 1(b)) produced by OLSON company. The frequency of the ultrasonic wave transducer was 54 kHz, with test response time of 0.01 ms.

2.2 Ultrasonic testing

The changes of ultrasonic wave velocity can reflect the inner damage of materials as the ultrasonic wave velocity is related to the microstructure in materials. The decrease of cracks shows a good agreement with the increase during loading. So the microstructure changes in rock salt may be inferred from the decrease of ultrasonic wave velocity based on a damage value defined as (Kawamoto *et al.* 1988)

$$D=1-(V_{pd}/V_p)^2 \tag{1}$$

where D is the damage value of rock salt, V_p is the longitudinal wave velocity of specimens before loading, and V_{pd} is the longitudinal wave velocity of specimens after loading.

2.3 Test scheme design

During excavations of openings in salt, microcracks would generate and develop in the deviatoric stress fields in the surrounding salt of the opening. These microcracks may increase permeability of the surrounding salt and therefore can increase fluid flow if interstitial gas is present. Excavation relaxes tensile stresses and allows compressive stresses in situ to close excavation-induced fractures enabling them to heal. Here, crack healing is the process whereby the normal salt lattice bonds are reestablished and the material strength is regained at the site of the healed crack. However, if the salt samples were badly damaged, it would be hard to recover. If crack closure is a prerequisite to crack healing, what will happen in the absence of stress condition? This paper aimed to study the relationship between damage degree and self-healing capacity under room temperature with no compression stress.

UPV was measured first on intact specimens, then after initial damage induced by uniaxial compression. The load stress was estimated at about 20, 30, 40 MPa according to the average uniaxial peak strength (45.23 MPa) of cuboid salt specimens. After that, the UPV of all loaded specimens were recorded again. And the measure direction was perpendicular to the loading direction because the majority of crack propagation directions are parallel to the loading direction. Generally analyzing the change of wave velocity perpendicular to the loading direction can better reflect the cracks development of the samples. All damaged salt specimens were placed into the self-healing environment with constant temperatures (room temperature) and no stress. The wave velocity of the damaged rock salts in self-healing environments was measured in fixed time period (recovery 0, 1, 3, 7, 10, 15, 21, and 30 days). Then we can analyze the self-healing characteristics of the specimens based on the wave velocity variation.

3. Results and discussion

The stress-induced initial damage of salt specimens can still recover under normal temperature and no stress environment. The microcracks developed gradually under the uniaxial loading stress, and continued to expand and connect into larger crack zones as loading stress increases. Although the specimens undergo the same loading stress, different initial UPV occurred on the specimens, as shown in Fig. 2. The UPV tests results of the damaged salt specimens during recovery period are shown in Figure 2. The UPV obtained from ultrasonic transmission method gives information about microstructures properties of materials, such as porosity, microcracks distribution and density. The UPV values of the damaged salt specimens increase quickly during first 9 days recovery except the very low damage-level salt specimens. And then the wave velocity increase very slowly until to stabilize. The UPV values of salt specimens will tend to stabilize after about 25 days of recovery. UPV values increasing showed that some parts of the internal microcracks are closed by strain recovery and recrystallization. However, some larger cracks were hard to recover only by strain recovery or recrystallization with no stress. The recovery period of damaged rock salt was a long-term and slowly procedure. In the test, the recovery of damaged salt keeps increasing for the first nine days of recovery due to the elastic energy recovery. The increase in recovery after this initial period is very slowly during the next 21 days, even more time.

The salt specimens underwent uniaxial compression which produces a large number of microcracks, and then some cracks appear with a certain amount of closure after stress relief. This corresponding strain recovery along with better coupling between the specimen and platens caused the wave velocity increase within the first 9 days. When the strain recovery is completed, the damaged specimen needs to increase compress stress or recrystallize to close the internal crack.

Wave velocity over time at cracks closure or healing appear to be damage-level dependent. The dependence of velocity on damage level follows from the data recorded during recovery. Examination of the data shown in Fig. 2 shows that the change in velocity increase in high damage level is more constant with increasing axial strain (or damage level) than in low damage level. When the process of crack development reverses during healing, the change in velocity with decreasing damage level should not be substantially different or specimens of high and low damage level.

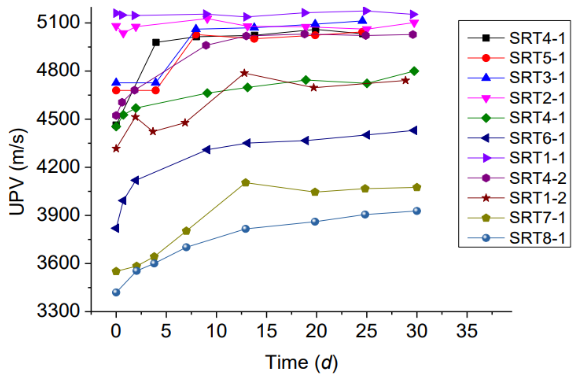


Fig. 2 Ultrasonic pulse velocity VS time curves of damaged salt specimens at room temperature and no stress condition

Table 1 Results of damage recovery test

Number	Recovery conditions	Damage value (D)			Recovery value (H)	Recovery percentage (%)
		Initial D_0	9d recovery	30d recovery D_{fin}		
SRT1-1	Room temperature without stress	0.06	0.06	0.06	0	0
SRT1-2		0.32	0.17	0.15	0.17	53.1
SRT2-1		0.13	0.13	0.13	0.01	7.7
SRT2-2		0.31	0.24	0.2	0.11	35.5
SRT2-3		0.52	0.39	0.36	0.16	30.7
SRT3-1		0.19	0.18	0.17	0.02	10.5
SRT3-2		0.35	0.18	0.18	0.17	48.6
SRT4-1		0.23	0.17	0.11	0.04	17.3
SRT4-2		0.28	0.17	0.16	0.12	42.8
SRT5-1		0.2	0.17	0.16	0.04	20.0
SRT6-1		0.38	0.29	0.26	0.12	31.6
SRT7-1		0.58	0.5	0.45	0.13	22.4
SRT8-1		0.61	0.55	0.5	0.11	18.0

Notes: Where Recovery value $H = D_0 - D_{fin}$ .and recovery percentage = $(D_0 - D_{fin})/D_0$, D_0 is the initial damage value; D_{fin} is the final damage value after finishing the recovery test

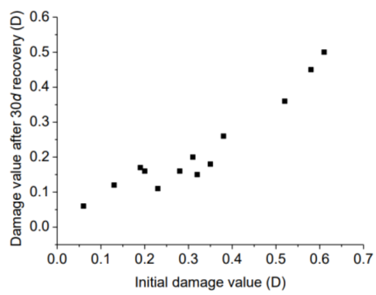


Fig. 3 Value of stress-induced initial damage vs. value of damage in stable stage

The above analysis shows that the wave velocity is affected by crack development. Wave velocity attenuation is a sensitive indicator for the damage-level, and it is very sensitive during the first 9 days due to cracks closure changed significantly. Wave velocity attenuation is caused

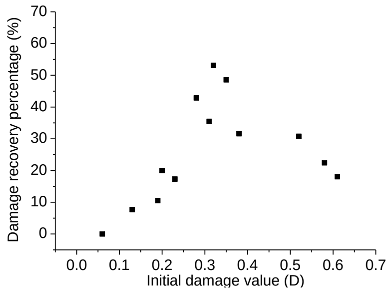
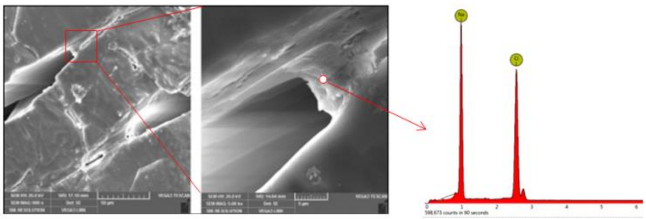


Fig. 4 Value of stress-induced initial damage vs. total value of damage recovery



(a) SEM pictures (b) Energy spectrum

Fig. 5 The microcrack tip were partly recrystallized under the self-healing test

by both scattering of energy at crack surfaces and frictional dissipation (Chen *et al.* 2013). Scattering is the reflection of energy off crack surfaces due to acoustic impedance mismatch. Frictional dissipation occurs when a seismic wave causes sliding along crack surfaces and grain boundaries. Frictional dissipation phenomenon did not occur in our recovery test for the no stress condition. Dissipation is affected by cracks which are slightly open. If cracks are tightly shut, a seismic wave will not provide enough force to overcome friction and cause slippage. If cracks are wide open, crack walls can vibrate with no loss on behalf of friction, and therefore no dissipation.

In Table 1, the UPV change is represented by the damage degree as expressed by Eq. (1). The initial damage, final damage (30 d), recovery value and recovery percentage of the rock salts are given in Table 1.

The degrees of initial damage are different with the same loaded pressure due to the differences of the structure or crystalline form in the specimens. In this paper we are mainly concerned about the difference of initial damage affected on self-healing and the load stress is irrespective. Fig. 3 shows that the higher value of initial damage exhibits greater final damage at the same recovery conditions. The final damage value almost linearly increases as the initial damage increases. However, recovery degree increased at the beginning, and turned to decrease when the initial damage reached the threshold. The relationship between recovery percentage and initial damage degree in Fig. 4 shows that the damaged rock salt specimen recovery exists threshold value. And it is difficult to recover when the damage reached the threshold damage value.

The damage recovery of rock salt samples mainly relies on strain recovery and recrystallization when there is no stress in the recovery process. To close and heal quickly, rock salt crack walls must return near to their original geometries so that they can touch and knot. For the

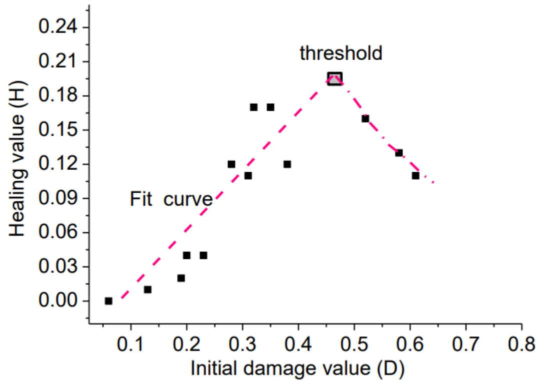


Fig. 6 Empirical relation between damage variable D and healing variable H

specimens with higher levels of damage, crack surfaces may become misplaced and may not contact immediately upon pressurization. Meanwhile, debris can fall into the crack and keep it open. Crack connected network in the specimens with low damage level are not as fully developed as those in specimens whit high damage levels. Damaged zones with many large cracks merely by elastic deformation closure, which means the recovery was difficult in no stress condition.

On the other hand, larger cracks could not be healed by crystal recrystallization recovery because a recovery condition that can bind salt grains together is necessary. During the recovery process, the cracks that have been caused by deformation are the first to close. Then the recrystallization healing could occur whereby grain boundary migration of grain boundaries overgrows the crack (Desbois *et al.* 2012b). Therefore, the rock salt damage will recover difficultly if the initial damage is too serious. It means there is a threshold damage value for the healing effect at the same recovery condition.

The microstructural investigation of the healed samples was made to observe the presence of grain growth (Fig. 5). According the SEM test, we found that the damaged rock salt samples with microcracks were healed due to the recrystallization under the suitable condition. We tested the new gains with Energy spectrum technology and just found the Na and Cl element, which means the new grains were pure rock salt. The new salt grains just appeared at a cracks tip, but they are hard to fill the large cracks.

4. The relationship between damage degree and self-healing

In the case of the coupled damage-healing process, one may use the physical relation between the damage and healing processes or introduce an empirical relation to define the governing relation between the healing and damage variables. We can define the heal variable (H) as $H=D_0-D_{fin}$. In this paper the heal variable include cracks closure and recrystallization.

For instance, the rock salt materials containing microencapsulated healing agent, one may find a relation between the introduced damage into the system and the diffused healing agent into the microcracks. Here, an empirical function is introduced which relates the damage variable D to the healing variable H as follows

$$H = \begin{cases} aD & if \ \ Z(D) > aD, \ \ a \in (0\sim1) \\ \alpha e^{-\beta_1 D+\beta_2} & if \ \ Z(D) \le aD, \ \ a \in (0\sim1) \end{cases} \tag{2}$$

and $$Z(D) = \alpha e^{-\beta_1 D+\beta_2}$$

where α, β_1, β_2, a are the material-dependent constants which may represent the physical characterization and the effectiveness of the healing agent. Eq. (2) is depicted in Fig. 6. The healing is assumed to recover all damages at the initial stages of the damage process. However, with the increase of introduced damage into the system, the effectiveness of the healing process is reduced.

5. Conclusions

Based on the analysis of the ultrasonic pulse velocity (UPV) variations for different initial stress-damaged rock salts during self-healing experiments, the self-healing effect of damaged salt specimens was analyzed during the recovery period of about 30 days.

The results suggest that both initial damage and time are important factors for the effectiveness of healing.

• The damaged rock salt specimens with difference initial damage induced by uniaxial compression could be recovered, but it was hard to heal completely. In this test, the damage recovers quickly in the first 9 days, and then the healing proceeds slowly until stabilizes gradually. The hydraulic conductivity for all salt fractures decreases with increasing applied pressure and time. This implies that fracture healing is accompanied by facture c1osure.

• Both processes are time-dependent. Some microcracks can be healed by recrystallization under suitable condition, but this process also influences recovery time. Anyway more stress-induced initial damage results in more internal macrocracks or connected microcracks, which results in a more difficult recovery. The healing is assumed to recover all damages at the initial stages of the damage process. However, it is found that a damage threshold exists for salt cracks healing. When the damage degree exceeds the threshold, the self-healing ratio is decreased with the increase in damage degree.

It is not clear, however, that what kind of damage of rock salt can be healed more effectively under recovery condition. This is because the recovery conditions are applied on the saw-cut fractures and with relatively low temperature. Since all fractures tested here are well mated, the impact of facture roughness cannot be truly assessed. More testing is needed to insure the mathematical relationship between the healing effectiveness and the initial damage. For the healing assessment method, a direct tension test could be used to minimize the impact of the stress gradient induced along the facture plane. From the results obtained here, it can be postulated that a complete healing of salt fractures is possible under preferable conditions (stress state, time, temperature, purity, crystal orientation, etc.).

Acknowledgements

This work was supported and financed by the Natural Science Fund (No. 51304256, 51604044, 41672292 and 51574048), Chongqing University Postgraduates' Innovation Project, Chongqing science and technology commission (Grant No. cstc2015jcyjA90011), the Fundamental Research Funds for the Central Universities (No. 106112016CDJZR245518), which are all greatly appreciated.

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