MESO-FAILURE MECHANISM OF ROCK UNDER THERMAL-MECHANICAL COUPLING EFFECTS

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ABSTRACT

The meso-failure mechanism of rock under thermal-mechanical coupling effects has been investigated in-situ through Scanning Electron Microscopy (SEM). Some failure phenomena, such as brittle fracture of sandstone could occur in two places, different minerals bear independently, brittle cracks of sandstone under thermal-mechanical coupling effects can also bifurcate, had clearly been reported. A lot of micro-cracks or branch cracks near the main crack had been observed when the test temperature was below 100 °C. However, when the temperature was over 125!, thermal activation of sandstone internal local zone would increase, and the ability to resist deformation and coordination has been strengthened. In addition, there were few cracks or secondary cracks near the main crack. From 25°C to 300°C, fracture toughness of sandstone at meso-scale. With the increasing of temperature, sandstone failure mechanism will transfer from brittle to ductility mechanism. Sandstone failure was affected by many fracture mechanism, such as transgranular fracture, intracrystalline fracture and mixed mode fracture. The deformation field of sandstone, which was obtained through SEM micro-measurement, was measured using Digital Speckle Correlation Method (DSCM).

Keywords: temperature, sandstone, meso failure mechanism, critical temperature, fracture toughness, DSCM

1. INTRODUCTION

The effects of temperature on the mechanical behaviour of rock at macro scale have been widely investigated experimentally [1-3]. It's of great importance to study the deformation and failure of the rock influenced by temperature. The influence of temperature on mechanical behaviors of rock is widely discussed at home and abroad, but researchers mainly focus on the macroscopic experimental study at laboratory scale [4-11], the theoretical model and numerical calculation method are developed for numerical analysis [12-15]. The influence of temperature on rock failure strain and failure angle is systemically concluded (Perterson et al.), the results show the angle between shear failure plane and axial load increases as temperature increases, but the trend is not very obvious. Wong [2] concluded the experiment results of 9 different types of rock, and found that the rock strength decreases as temperature increased at laboratory scale, this is confirmed by experiments. Zhang [11] studied the effect of temperature on fracture toughness of granite by three point bending test. The DECOVALEX [12, 13] is the most representative one among the studies about heat-fluid-tress coupling problem, and many meaningful results were gained. These studies mostly focused on the development of corresponding numerical calculation model or large scale field in-situ tests. Recently, the mesoscopic thermal-mechanical (TM Model) coupling model has been developed, and the microstructure damage of rock material and the evolution mechanism of material mechanical properties were discussed. Because this study is based on the premise that the mechanical behaviors of rock material follow certain distribution, it need further research when discussing the actual failure mechanism. Scholars have studied the rock meso-deformation failure mechanism, these studies focused on the evolution process of the interior micro-cracks and micro-voids in rock [14-16]. The effect of temperature was less discussed.

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Generally speaking, the studies about rock failure mechanism in the effect of temperature are presented from the macroscopic view, while due to the limited experimental condition, the effect of temperature is not considered in the studies about rock failure mechanism at micro meso-scale. More importantly, the process of rock deformation failure under applied load following the effect of temperature can't be observed in most experiments. Actually, the effect of temperature on the rock mechanical properties is mainly found in the rock mineral composition and microstructure, so as to affect the mineral property, internal bonding state, microcracks and holes of the rock. These effects are finally presented from the stress-strain relationship in the macroscopic view. So the failure behavior in the macroscopic view is essentially limited by definitive mechanical process at micro- meso scale, and it is possible to study the rock failure mechanism from the micro-meso scale. Then the rock macro failure behavior can be studied. Based on this, this paper studies sandstone failure through the real-time observation by SEM, and the deformation field is measured and presented by DSCM.

2. EXPERIMENTAL CONDITIONS

The sandstone sample is fine siltstone which is gained from the 1st Coal Mine of Pingdingshan Coal mine. The specific size of the processed specimen is shown in Fig. 1. For observing and capturing the sandstone failure process, a double edge notch is precast in the middle part of the specimen. The notch is about 0.2mm deep. Due to the poor electrical conductivity of rock, KYKY SBC-12 ion splash instrument is used to sputter-coat with gold for 1.5-2 minutes. The test is completed by the SEM high temperature fatigue experiment system of State Key Laboratory of Coal Resources and Safe Mining, China University of Mining and Technology, Beijing. The experimental procedure is firstly increasing the temperature and then keeping the constant temperature to load, the heating rate is 5!/min. In the process, the sample is loaded until the tensile failure happens; the load orientation is shown by the arrows in Fig. 1. The displacement is controlled when loading, and the loading rate is 10⁻⁴ mm/s. The test system can automatically record the load and displacement of the stroke apparatus. In the loading process, the evolution of the surface morphology, failure pattern and crack growth morphology can be observed and photographed in situ by SEM.

3. THE STUDY OF THE MESO-FAILURE PROCESS OF SANDSTONE UNDER TEMPERATURE EFFECT [6]

One directional tensile test is carried out under the temperature of room temperature (25 °C), 30 °C, 35 °C, 40 °C, 45 °C, 50 °C, 100 °C, 125 °C, 150 °C, 175 °C, 200 °C, 250 °C and 300 °C. At each temperature point, three successful tests are carried out. For example, 150 °C-3 stands for the third specimen under 150 °C.

3.1. Failure Tests with the Temperature Below 100 °C

For the specimen 25 °C-3, the micro crack appears when the load is 12 N, as shown in Fig. 2 (a). The cracks spread continually with the increasing load. The specimen ruptures when the load is 13.3 N, as shown in Fig. 2 (b). For the cracks are beyond the visual scope of SEM, the full view of the rupture can't be observed, the local destruction process is shown in Fig. 2(c). The load variation range is small from the appearance of the micro crack to the rupture, and near the main crack, there are many branch cracks, so this is a typical brittle rupture.



Figure 1: The Size and Shape of Double Edge Notched Specimen



(c) local enlarged image (×100)

Figure 2: Failure Process and Local Enlarged Image on Specimen 25!-3

For the specimen 30 °C-2, as the load increases, the crack firstly appears at the precast notch, this is because the stress concentration phenomenon is very obvious at the precast notch. When the load increases to 12.5 N, cracks appear near the upper precast notch, which is shown in Fig. 3(a). When the load reaches its limit, the micro cracks on the surface of the specimen begin to interpenetrate and finally the specimen ruptures. It is illustrated in Fig. 3(b). Therefore, both the locations of the initial crack and the micro crack on the surface affect the rupture of the specimen. It is shown in the SEM photos that sometimes the crack spread by damaging the mineral particles, and sometimes the particles are bypassed. The rough calculation shows there are 60 to 75 percent rupture appears on the boundary of the particles and the cement, while only a small portion of the rupture goes through the particles. According to the fractal theory, this is mainly because that intracrystalline fracture would consume less energy than transgranular fracture.

For the specimen 50 °C-1, as the load increase, the cracks dispersively attribute at the surface. When the load is 13.4N, the micro cracks begin to spread towards the main crack, and some of the cracks begin to bifurcate, as shown in Fig. 4(a). With the increasing load, the micro cracks converge and form a visible main crack, this leads to the stress field unloading of the local zone near the main crack. The branch cracks and secondary cracks then stop spreading. When the load is 18.8 N, the cracks converge and form a visible main crack that perpendicular to the direction of tensile stress, then the specimen ruptures, as shown in Fig. 4(b). The experiment for specimen 50!-1 shows that, at the beginning of the experiment, the cracks distribute randomly on the surface of the specimen, as the load increases, micro cracks aggregate and spread along the direction perpendicular to the tensile stress. Finally, the macro main crack is formed. Because sandstone contains the quartz, dolomite and calcite, the extension of crack has the tortuous path. There are also many micro cracks that do not spread or spread a little, which can not lead to form the main crack. Because of the stress concentration near the precast notch, the cracks spread towards the two



(a) 12.5 N (×35)

(b) 12 8 N (×35)





(b) 18.8N (×50)

Figure 4: SEM Images of Surface Cracks on Specimen 50!-1

stress concentration spots. The tow stress concentration spots are like two attractors. They affect the spread path fundamentally. In this case, stress concentration plays a dominate role comparing with the micro defects when the main crack penetrating the two precast notches.

3.2. Failure Tests with the Temperature Above 100 °C

For the specimen 150 °C-1, the surface of the specimen has little change in the process of the load increasing to 25.9 N, as shown in Fig. 5(a). When the load increases to 47.6 N, the specimen suddenly ruptures, this means sandstone exhibits brittleness at this time. What's interesting, the ruptures happen at two locations. In the first case, the rupture begins at location A and goes through the upper precast notch. In another case, the rupture begins as location B which is about 2.07 mm away from the lower precast notch, and it bifurcates when spreading to the center position of the specimen, as shown in Fig. 5(b). The results of the experiment show that brittle fracture can happen at two different locations simultaneously, that is to say the fracture of the rock is not necessarily dominated by one main crack, and maybe there are two or more main cracks. This also shows that various minerals or minerals at different cross sections bear the load as the time changes. The deformation field of this experiment is calculated and analyzed by digital speckle correlation method (DSCM) in the following part.

For the specimen 150 °C-2, when the load is 45.8 N, micro rupture happens near the lower notch, actually it's on one mineral particle, as shown in Fig. 6(a), this the sign of the beginning of deterioration; According to the experimental equipment record, when the load reaches its maximum 46.7 N, the specimen doesn't lose its bearing capacity. Six load points are still recorded, they are 46.5 N, 46.1 N, 46 N, 45.6 N, 44 N and 45 N, the duration is

about 28s (the data is recorded every 4s), when the load is 45 N, rupture happens. It can be observed that for the brittle material sandstone, several fracture mechanisms work. Fig. 6(b) shows that the rupture directly splits the mineral particle at the lower position, and most ruptures are along the edge of the particles at the upper position. It is regarded that the meso-mechanism of the through-particle-rupture and along-particle-rupture work together. The crack bifurcation phenomenon of brittle rupture can also been observed clearly. The macro crack appears in the middle-upper part at the beginning, and then it spreads downwards. It suddenly bifurcates when it's close to the lower notch, one direction is towards the location already been deteriorated, another is towards other week locations.

For the specimen 175 °C-2, when the load is 25.2 N, the cracks appear near the lower notch. Firstly the cracks appear at the clay cement, and then they spread through a large mineral particle, as shown in Fig. 7(b). As the load increases to 34.5 N, the cracks spread furthermore. When the load increases to 37.9 N, the cracks extent throughout the specimen and finally the specimen ruptures, as shown in Fig. 7(d). The ruptures mainly happen on the clay cement and the junction of the particles and cements (68%-80%), the rest happen on the mineral particles. This is mainly because that intracrystalline fracture would consume less energy than transgranular fracture, the transgranular fracture is more easily to happen [17].

For the specimen 200 °C-1, in the loading process, the mineral particles near the lower notch begin to rupture, these particles maybe the main matrix to bear the load, as shown in the lower part of Fig. 8(a). After the main bearing particles rupture, the rock bridge (mainly clay cements and other particles) between them begin to converge and form a main crack, the central part in Fig. 8(a) is the intersection of the two cracks; after the main crack is formed, the cracks continually spread upwards, and they gradually spread towards the stress concentration locations,



(a) 25.9 N

(b) 47.6 N





(b) 45 N

and finally they interpenetrate with the upper notch which leads to the general rupture. The main crack goes through the precast notch, which is about 0.473 mm away from the center of the lower precast notch. "3" shape cracks appear at local area of the rupture, as shown in Fig. 8(b). The formation of the cracks could be ascribed to the following reasons: the first one is that the local stress concentration phenomenon is very in the process of crack spreading, the second one is that the cracks change the spread direction when meeting the particle interface or hard mineral particles; The third one is that two cracks on different planes join together by splitting the week part between them, this one could be the main reason to form the cracks of this shape.

For the specimen 250 °C-1, when the load is 23.8 N, a few bearing particles begin to rupture. When the load increases to 27.5 N, the specimen ruptures, as shown in Fig.9 (a). When the load increases to 22.4 N, the second rupture happens at the upper part of the specimen, as shown in Fig. 9(b). The relationship between the displacement of the stroke apparatus and load is shown in Fig. 9(c), this Figure shows that the load changes. The first rupture spreads from bottom to top, while the second rupture spreads from top to bottom, and the two ruptures are not on the same plane. Because there is a time difference between the two spreads, when the second rupture happens, the two spread cracks exceed a specific distance while converging due to the high speed. But later the week rock bridge is split, and the two cracks converge which leads to the rupture. Because sandstone contains various mineral composition, the crack stops spreading after a certain distance, as the load increasing, the sandstone begin to rupture from another



Figure 7: Failure Process of Specimen 175!-2 (×35)



(a) crack convergence (×100)

(b) the shape of "3" crack $(\times 350)$



Figure 9: Failure Process and Load-displacement Curve of Specimen 250!-1 (×35)

location. It shows that the minerals on different cross sections may bear the load independently under different stress conditions.

The rupture of specimen 250 °C-1 and 150 °C-1 is very similar. The phenomenon of the two experiments shows that when sandstone is imposed by tensile stress, it is not the whole mineral particles on the cross section bear the load but only a part of the mineral particles and clay cements. After this part of the minerals ruptures, the rest of the mineral particles and clay cements begin to bear the load, the two parts are just as parallel connected on the same cross section, and play the main role in bearing the load. This is very similar with the specimen 150!-1. It is also proved that the study of damage behavior by parallel levers conform to the reality.

For the specimen 300 °C-2, when the load increases to 37.7 N, the cracks along the particles appear on the interface of a triangular shape mineral particle and clay cements, which is indicated by the arrow in Fig. 10(a); As the load increasing, the cracks spread upwards, the whole specimen ruptures when the load is 38.9 N, and the main crack penetrates the two notches, as shown in Fig. 10(b).

4. FRACTURE TOUGHNESS OF SANDSTONE UNDER DIFFERENT TEMPERATURE

For the analysis of the effect of temperature on fracture toughness of the sandstone characteristic, the approximate calculation of the fracture toughness of the sandstone specimen with double edge notches by fracture mechanics is presented. There is no mature standard in the calculation of rock fracture toughness, so the calculation formula



Figure 10: Failure Process of Specimen 300!-1 (×35)

based on some metal materials is used. The main focus is on the effect of temperature, so the analysis results gained through the same formula are not affected. The fracture toughness calculation formula for the double edge notches tensile specimen is as follows:

$$K_{\rm Lc} = \frac{Pa^{1/2}}{BW} \left[1.98 + 0.36 \left(\frac{2a}{W}\right) - 2.12 \left(\frac{2a}{W}\right)^2 + 3.42 \left(\frac{2a}{W}\right)^3 \right]$$
(1)

where a is the length of the precast crack, B is the thickness of the specimen, W is the width of the specimen, P is the fracture load.

The fracture toughness of 37 specimens under different temperature is shown in Fig. 11. The Figure reveals sandstone fracture toughness is higher when the temperature is above 100! than it is below 100 °C.



Figure 11: Fracture Toughness of Sandstone under Different Temperatures

5. ANALYSIS AND DISCUSSION

The effective loading area of the cross section through the precast double edge notches is the smallest, and the precast notches can change the stress field near it, which leads to the stress concentration. Under external loadings, the area near the notches reaches ultimate failure first. From theoretical point of view, sandstone is more easily to rupture near the precast notches. But the experiments show the different results. In the experiments when the temperature is below 100 °C, the rupture locations have highly randomicity, many of them do not happen at the notches, some of them are even far away from the notches. When the temperature is above 100 °C, most of the ruptures happen at the precast notches or near them. In the experiments below 100 °C, some branch cracks or secondary cracks are dispersively distributed on the surface, it can be observed in the experiments when the temperature is 25 °C, 30 °C, 40 °C and 50 °C. Because of the low temperature, the brittle mechanism plays a dominate role, and the cracks have a highly randomicity when spreading. At the beginning, the micro cracks in sandstone have the tendency to spread towards the cross section penetrating through the notches. Because the brittle mechanism plays the dominate role, and the spread of the cracks is catastrophic and gusty, together with the heterogeneity of the rock, these cracks can not control their spread, the distribution of the rupture locations have great discreteness. To some extent, this shows that when the temperature is below 100 °C, the uniform deformation ability is very poor, and the brittle mechanism plays a dominate role. As the temperature increasing above 100!, the thermal dilation of mineral particles on the surface begins, and the brittle mechanism decreases to a certain extent, while the ductile mechanism enhances, this is because of the enhancement of the local ductile characteristic and the ability to resist deformation and coordination, more details see reference [18]. Actually, when the temperature is above 100 °C, there is few branch cracks appear near the main crack, to some extent this means the transition of brittle mechanism to ductile mechanism or the tendency to transit as the temperature increasing. With the external load imposed on the specimen, the mineral particles and cements can well coordinate displacement between them. So the uniform deformation ability is enhanced when the temperature is very high.

The experiments show the fracture toughness is highly enhanced after the temperature is above 125 °C, as shown in Fig. 11, actually it is not accidental. Comparing the depth of all the precast notches, it is found that generally speaking, precast notches of the three specimens under 150 °C are slightly large, but the fracture toughness is higher. For the specimen under 250, their notches is almost the same as the ones under 150 °C, but the fracture toughness is higher than the specimen under low temperature, so the temperature has a great impact on the fracture toughness. The notches depth of the three specimens under 250 °C is smaller than that of the ones under 150 °C, but the fracture toughness is of statistically small, it is proved again that 150 °C is the temperature to reach the extreme value. Between 25 °C and 150 °C, the fracture toughness decreases, this is because of the thermal cracking [19]. Inspite of the temperature, the path of the crack is toutuors. Statistical results show that about 60-80% of the ruptures are along the particles, while only 20-40% ruptures go through the particles. According to the fractal mechanic theory [17], this is mainly because that intracrystalline fracture would consume less energy than transgranular fracture.

Many SEM photos of the failure process have been given, the deformation of the rock can not be observed by naked eye, but local deformation has already appeared before failure, SEM can record this information, so how to describe the deformation field is an important task. Through related image processing method, this paper measures the meso deformation filed under SEM by Digital Speckle Correlation Method. The basic principles of DSCM have been discussed in [6], the process is to get the in-plane displacement component and gradient through the photos in different deformation or different deformation time. The process of distinguishing the deformation is: collect the two digital photos before and after the deformation, and then define the small plot before deformation of a specimen sub area. The plot in the photo after the deformation is defined as target sub area correspondently. The deformation of can be extracted through the one-to-one correspondence between the target sub area and specimen sub area. The SEM and DSCM are integrated to measure the deformation field under SEM.

For the study of the local deformation field, the square zone in Fig. 5 (a) was selected to calculate, the SEM photo under the load of 25.9 N. In this case, the results of the calculated displacement field and the calculation datum. The deformation field with a load difference of 21.7 N is calculated, the results of displacement field is shown in Fig. 12 and Fig. 13.

There are two step indexes in the deformation field of sandstone, which can be seen clearly in the displacement field of the axis tensile orientation in Fig. 12. This means ruptures happen at two different locations, and the

discontinuity of displacement at the cracks is presented, the deformation near the cracks is very large, and the largest horizontal displacement is about 2 im. Two rupture locations can also be observed in displacement contour maps, as shown in Fig.13. The rotation phenomenon and inhomogeneous deformation at local zones are observed.

It is indicated that DSCM can measure continuously of the local failure process of rock. Both the graph of displacement field and displacement vector can show the displacement field invisible at macro scale. This provides a new research approach for the study of deformation field of rock at micro/meso scale.



Figure 12: Displacement Field Maps of $\Delta P = 21.7$ N (Unit: μ m)

Figure 13: Displacement Contour Maps of $\Delta P = 21.7N$

6. CONCLUSIONS

The meso-failure progress of sandstone under temperature effect is observed in situ through Scanning Electron Microscopy (SEM). It is considered that for sandstone specimen at meso scale, temperature has a great impact on the deformation, the specific conclusions are as follows:

The meso-failure progress of sandstone under temperature effect is observed in situ, and some new experimental phenomena are found, for example, brittle fracture of sandstone could occur in two places or more, different minerals in different cross sections or even in the same cross section can bear independently. In the process of loading, not all the minerals bear. At the beginning, only a portion of the minerals bear until they rupture, and then the rest minerals on the cross section or the minerals on other cross sections begin to bear until rupture; Brittle cracks of sandstone under thermal-mechanical coupling effects can bifurcate, have clearly been observed. 150! is the threshold temperature of fracture toughness of sandstone at meso scale: From 25°C to 100°C, fracture toughness of sandstone changes little or changes gradually; From 100°C to 150°C, the fracture toughness increases and reaches the extreme value; From 150 °C to higher temperature (such as 200°C, 250°C and 300°C). Because of thermal cracking, the fracture toughness decreased. The failure process of 33 sandstone specimens was observed in situ. It showed that the temperature had a great impact on loacal deformation rule When the temperature is $low(25 \sim 100 \text{ °C})$, there were branch cracks or secondary cracks on the surface, which means the intracrystalline fracture mechanism dominated; With the temperature increasing $(125 \sim 300^{\circ}C)$, because of thermal activation, the ductile characteristic of local zones in sandstone minerals was enhanced. The ability to resist deformation and coordination will be enhanced, there were few branch cracks or secondary cracks on the surface. The ductile mechanism works, and the temperature affected the fracture toughness. The intracrystalline fracture mechanism dominated statistically because that intracrystalline fracture would consume less energy than transgranular fracture. The deformation field of sandstone, which was obtained through SEM micro-measurement, was measured using Digital Speckle Correlation Method (DSCM). The local deformation of sandstone can well been measured, and the rotation phenomenon and inhomogeneous deformation at local areas were found. The paper indicates that DSCM can measure continuously of the local failure process of rock.

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