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Research of microcosmic mechanism of brittle-plastic transition for granite under high temperature

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Abstract

The effect of temperature on mechanical characteristics and behaviours of granite was analyzed by using experiments as scanning electron microscope(SEM), X-ray diffraction and acoustic emission(AE) and the micromechanism of brittle-plastic transition of granite under high temperature was discussed as well. SEM result shows that the fracture surface possesses the mixed-rupture characteristics of transcrystalline cracks, cleavage step, slip band and superficial dimple at about 800℃; X-ray diffraction shows crystal form of some crystals of granite has transformed; and AE strength decreases, but the duration increases at about 800℃. The residual plastic deformation of granite still releases serried acoustic emission especially after peak strength, which is uniformly with the variation of macromechanical characteristics of granite. The failure form of granite changes from abruptly brittle fracture to semi-brittle shear fracture gradually with the rise of temperature. The research findings provide valuable basic references for the rock thermodynamics problems in reposition of nuclear waste, practical gasification of coal and oil shale, deep resources mining, exploitation of geothermal resources and safety drilling as well as comprehensive utilization of coal seam gas.

Keywords: granite; acoustic emission (AE); X-ray; scanning electron microscope; brittle-plastic transition

1. Introduction

At present, major engineering problems, such as reposition of nuclear waste, deep resources mining, practical gasification of coal and oil shale, exploitation of geothermal resources, safety drilling and comprehensive utilization of coal seam gas, make the study of rock physical and mechanical properties under high temperature a very active research topic in rock mechanics field[1-4]. In the last few years, some investigations theoretically and experimentally have been carried out in this field and great achievements have been made. For example, Brede M[5] studied the efffect of temperature on material brittle-ductile transition, and found the brittle-ductile transition temperature increases with rise of loading rate. Alshayea N A [6] researched rock damage process under heating using acoustic emission, and measured fracture toughness KIC of Westerly granite from 20℃ to 50℃. An experimental study on the brittle-plastic transition in gabbro had been researched by Sang zu-nan[7]. The temperature of brittle-plastic transition is 700~900 ℃.The major factors that affect the brittle-plastic transition in the gabbro are temperature, confining pressure and strain rate.

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Because the granite has low penetrability, compact and high strength, so the temperature of its thermal fracturing and brittle-plastic transition is high. The temperature of granite studied is under 800℃ in former research, so the effective temperature of brittle-plastic transition can not be obtained, which influences the mechanism research of granite brittle-plastic transition [8-10]. So, this investigation focuses on the stress-strain behaviors and acoustic emission characteristics of granite under the action of temperature from normal temperature to 1300℃ were investigated. X-ray powder diffraction analysis on heated granite was studied, and fracture appearance was also scanned. In this paper, the key factor which causes sudden change of rock mechanical properties and the mechanism of brittle-plastic transition under high temperature was researched from mechanical characteristics and structure crystallography.

2. Experimental

2.1. Experimental method

In our investigation, the rock specimens are 50 mm long, with diameters of 25 mm. There were 45 specimens in total, divided into nine groups with 3 specimens to each group. First, the specimens of each group were heated to the temperature of 25, 50, 100, 200, 300, 400, 500, 600, 700, 800, 900, 1000, 1100, 1200 and 1300℃ separately. In each case, the temperature was kept constant for 20 min so that the specimens could be heated to the assigned value from the outside to the inside and then the specimens were taken out and cooled down in the air. The experiment used displacement control mode, and the loading rate is 0.0015mm/s. Stress-strain behavior was obtained and mechanical characteristics such as load, deformation, time and acoustic emission characteristics were also measured in the process of rock deformation and destruction after exposure to high temperatures. X-ray powder diffraction analysis on heated granite was studied, and fracture appearance was also scanned.

2.2. Experimental equipment

All the samples used in the lab experiments were taken from the production line of the plant. The feed, intermediate materials, products of the limestone and clinker systems and the coal feed were each sampled from conveyor belts or chutes. The ground coal and final cement products were sampled with a CY40 automatic powder sampler. Each sample was taken for a period of at least two hours to ensure that a representative sample had been collected. Sampling time intervals, and the weight of the subsamples, conformed to the national and coal industry regulations and standards.

3. Results and analysis

3.1. Stress-strain curves of granite after high temperature

Because the stress- strain curves of each rock group has a similar form of distribution, we just present some representative samples, as shown in Fig. 1.

From Fig. 1, we can draw two conclusions. 1) The strength decreases suddenly and the granite presents plastic and post-peak behavior after 800℃, which indicates that a phase change behavior of brittle-ductile transition appears around 800℃. 2) The experimental results also show that after high temperatures, the sample failure mode changes from abrupt instability to a gradual failure with an increase in temperature. The failure type is a brittle or semi brittle shear fracture below 800℃ and a semi ductile shear fracture at temperatures over 800℃, showing crystal plasticity deformation has occurred, which changes to plasticity at 1200℃. The strength has no obviously decrease before 800℃, the reason is that the rock is cooled down after heating, which recovered the brittleness of granite. So the mechanical behavior of rock shows breaking state, which is closely related to the alteration of rock crystal form and brittle-plastic transition.

The average of compressive strength and elastic modulus at different temperatures is seen as Table 1, which decreases with the rise of temperature.

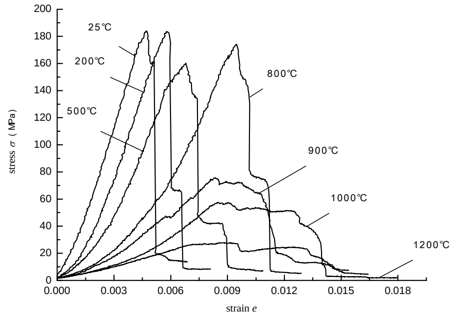


Fig. 1. Stress-strain curves of granite after high temperature

Table 1. Average of compressive strength and elastic modulus at different temperatures

Temperature (℃)	25	200	500	800	900	1000	1100	1200
Compressive strength d σc (MPa)	191.90	135.96	151.90	185.22	89.94	71.61	77.98	36.09
Elastic modulus E (GPa)	38.37	28.68	31.25	25.11	11.02	8.39	6.61	2.87

Rock failure form at different temperatures is shown in Fig.2.

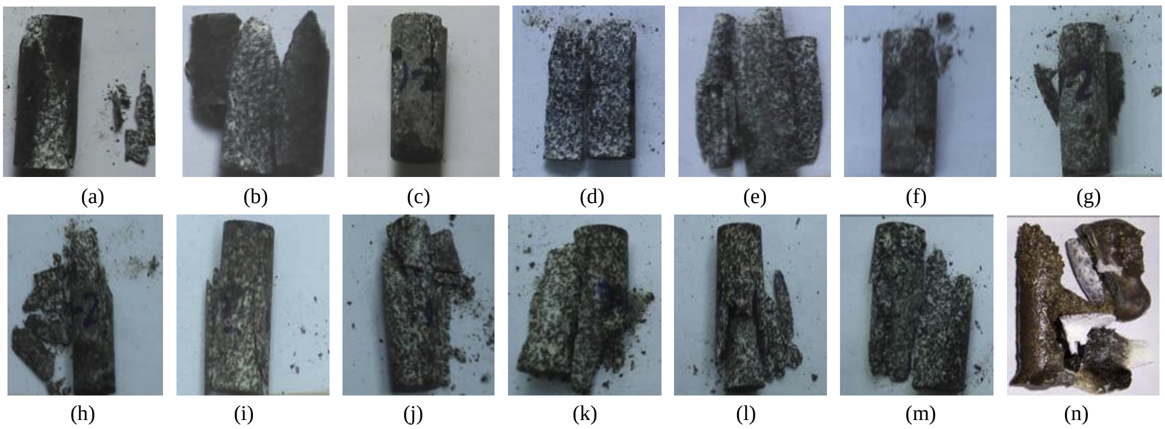
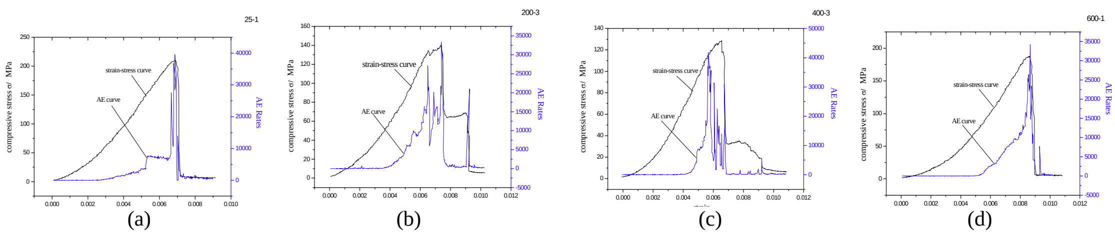


Fig. 2. Macro-fracture after different temperatures (a) 25℃; (b) 100℃; (c) 200℃; (d)300℃; (e)400℃; (f) 500℃; (g) 600℃; (h) 700℃; (i) 800℃; (j) 900℃; (k) 1000℃; (l) 1100℃; (m) 1200℃; (n) 1300℃

With the rising of temperature, failure form of granite changes from abruptly brittle fracture to gradually semi-brittle shear fracture. When temperature reaches 1300℃, the sample is almost in melting state.

3.2. Strain-stress curve and AE rates at different temperatures

AE characteristics were described using AE count rates, which can reflect the frequency of rock internal rupture. Relationship between strain-stress curve and AE rates of granite at different temperatures is shown as Fig.3.



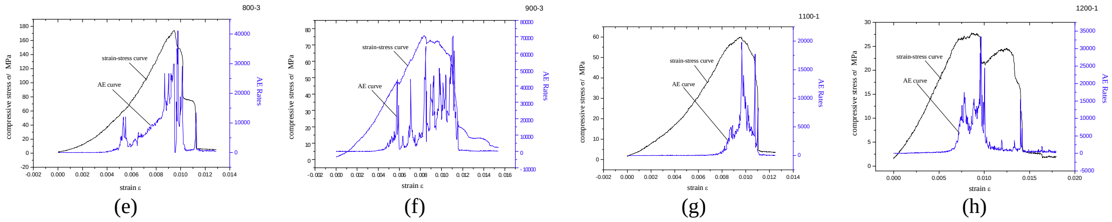


Fig. 3. Relationship between strain-stress curve and AE rates of granite at different temperatures; (a) 25℃; (b) 200℃; (c) 400℃; (d) 600℃; (e) 800℃; (f) 900℃; (g) 1100℃; (h) 1200℃

Strain-stress curve and AE rates of granite at different temperatures agree well, acoustic emission always occurs at the turning point of loading curve, the turning point means the mutation of energy, which is consistent with the AE is the internal energy release suddenly. AE strength decreases, but the duration increases about 800℃, the residual plastic deformation of granite still releases serried acoustic emission especially after peak strength, which is uniformly with the variation of macromechanical characteristics of granite.

3.3. X-ray diffraction experimental results and analysis

According to x-ray diffraction results, x-ray diffraction pattern of granite at different temperatures is shown as Fig. 4.

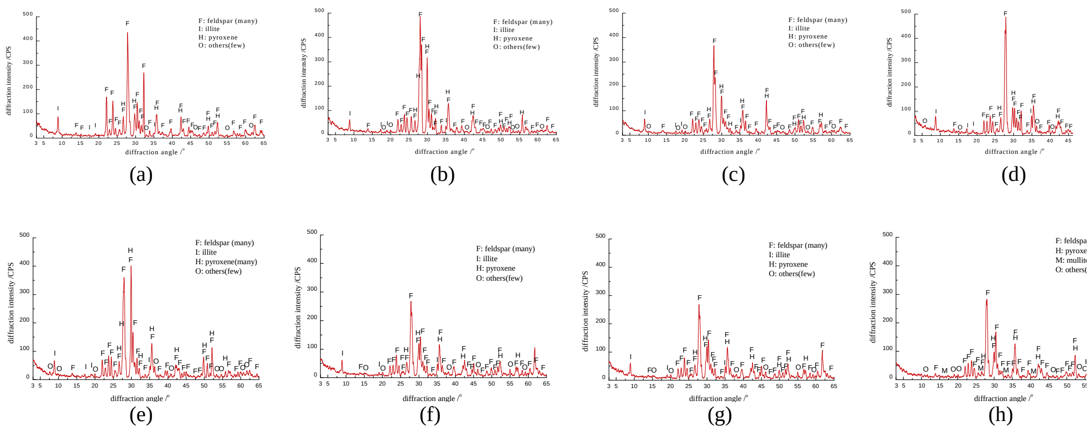


Fig. 4. X-ray diffraction pattern of granite at different temperatures (a) 25℃; (b) 100℃; (c) 200℃; (d) 300℃; (e) 500℃; (f) 800℃; (g) 1000℃; (h) 1200℃

The max diffraction intensity of granite principle ingredients at different temperatures is shown as Table 2.

The main composition of sample is feldspar, together with some illite, pyroxene and few other minerals. The feldspar contains mainly of anorthite and albite, with some potash feldspar. The results show that the diffraction information has no obvious variation below 800℃. The diffraction intensity of feldspar and pyroxene become lower and wider at 800℃, the diffraction intensity of feldspar decreases from 438CPS of normal temperature to 270CPS, and pyroxene from 402CPS of 500℃ to 146CPS. However, the chemical compositions of ultimate products have no variation, which indicates that the phase transition from crystalline to noncrystalline has occurred. It is because that the feldspar is the main composition of the sample, the chemical composition is aluminosilicate of Na, K and Ca. The differential thermal curve of feldspar appears endothermic valley, which is equivalent to polymorph transformation. It is the key factor which causes sudden change of rock mechanical properties over 800℃.

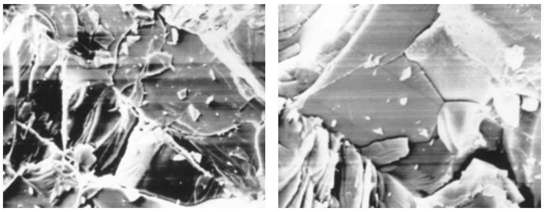
3.4. Analysis of granite fracture image with SEM

Granite is to typical brittle rock, which displays typical brittle failure under room and lower temperature. Failures begin to be transformed from brittle to plastic deformation when temperatures are over 800℃. Several representative pictures are presented in Fig.5.

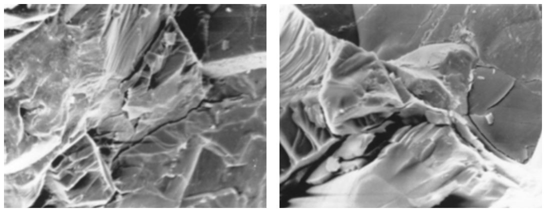
Table 2. Average of compressive strength and elastic modulus at different temperatures

Temperature/℃	25	50	100	200	300	500	800	1000	1200
Imax(feldspar)/CPS	438	390	487	363	488	402	270	271	283
Imax(pyroxene)/CPS	149	160	320	160	123	402	146	146	130
Imax(illite)/CPS	90	50	58	68	80	70	61	62	0
Imax(mullite)/CPS	0	0	0	0	0	0	0	0	34

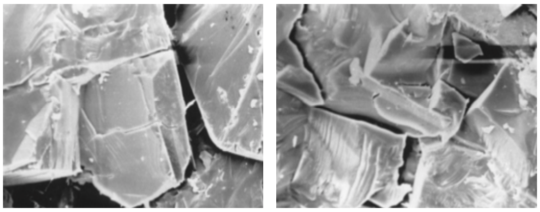
Analysis of fracture surface is the common effect of temperature and stress. Fracture surface of granite is smooth and fracture appearance is candy and river-shaped brittle tensile fracture at temperature lower than 800℃.The fracture surface possesses the mixed-rupture characteristics of transcrystalline cracks, cleavage step, slip band and superficial dimple. Brittle-plastic transition and crystal form transition have appeared at temperature higher than 800℃. The temperature of brittle-plastic transition can be deduced about 800℃.



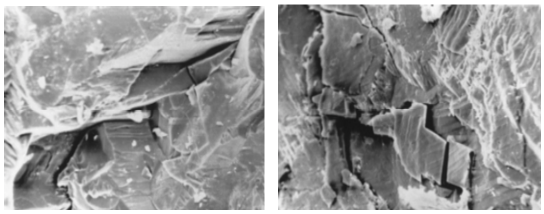
(a)



(b)



(c)



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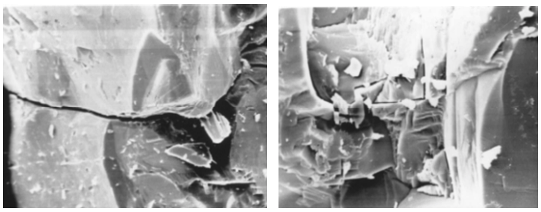

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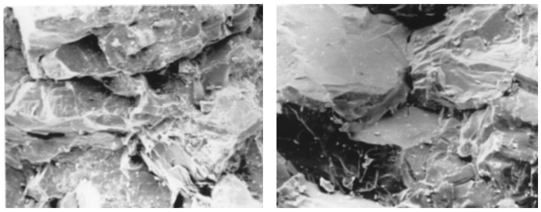
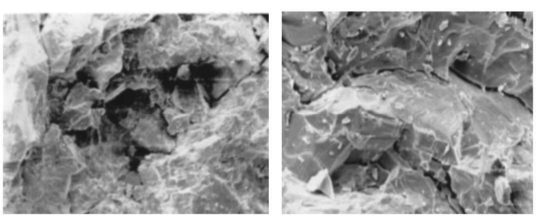
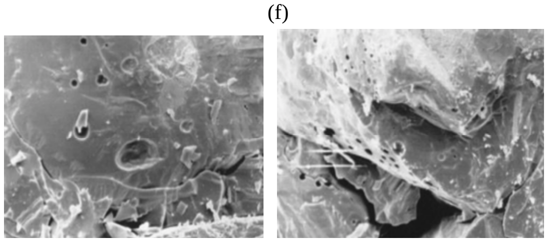


(f)



(g)



(h)

Fig. 5. Fracture image with SEM of granite at different temperatures (a) Candy pattern, River let pattern (25℃); (b) River let pattern (100℃); (c) Cleavage step, Intergranular fracture (200℃); (d) River let pattern, Cleavage step(300℃); (e)Cleavage step, Transgranular fracture(500℃); (f) Transcrystalline cracks, Slip band(800℃); (g)Rough crystal surface, Dimple , Thermal cracking(1000℃); (h)Dimple, Thermal melting, Micropore (1200℃)

4. Conclusions

1) The AE signal shows obviously brittle failure characteristics below 800℃. Over 800℃, AE strength decreases while the duration increases, the residual plastic deformation of granite still releases serried acoustic emission especially after peak strength, which is uniformly with the variation of macromechanical characteristics of granite.

2) The diffraction information has no obvious variation below 800℃.When temperature over 800℃, the diffraction information becomes lower and wider, which indicates phase deformation of brittle-ductile transition has appeared. It shows that the phase transition from crystalline to noncrystalline is a key factor which causes sudden change of rock mechanical properties under high temperature, while this point is usually ignored.

3) The fracture surface possesses the mixed-rupture characteristics of transcrystalline cracks, cleavage step, slip band, and superficial dimple. Brittle-plastic transition and crystal form transition have appeared at temperature higher than 800℃. The temperature of brittle-plastic transition can be deduced about 800℃.

Acknowledgements

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