

## Research Article

### Mineral Compositions and Micro-Structural of Epoxy-Repaired Rock Revealed by X-ray Diffraction and Scanning Electron Microscopy

<sup>1</sup>Wei Hu, <sup>2</sup>Xu Wu, <sup>1</sup>Jiawen Zhou, <sup>1</sup>Xingguo Yang and <sup>1</sup>Minghui Hao

<sup>1</sup>State Key Laboratory of Hydraulics and Mountain River Engineering, Sichuan University, Chengdu, 610065, China

<sup>2</sup>Sino Hydro Bureau 7 CO., Ltd., Sino Hydro Group Ltd., Chengdu, 610081, China

**Abstract:** In order to improve the mechanical properties of rock with lots of cracks, this study adopts electronic methods (X-ray diffraction and scanning electron microscopy) to determine the mineral compositions and micro structural characteristics of epoxy-repaired rock. Fractured rocks with low strength or high permeability may not be appropriate for dam foundation. The strength and durability of fractured rock are increased after chemical grouting. Test results show that, some of the cracks and voids are repaired by epoxy particles, resulted in the decreasing rock porosity. The proportion of epoxy resins is about 3.5%-5.5%, showing a discrete distribution and can not connect with each other.

**Keywords:** Epoxy-repaired rock, microstructural, mineral compositions, scanning electron microscopy, X-ray diffraction

## INTRODUCTION

Rock contains lots of cracks, the mechanical properties of rock are influenced by these cracks and the cracks may represent the total extent of the damage (Issa and Debs, 2009; Zhou *et al.*, 2012). Their significance depends on the type of structure, as well as the nature of the cracking. In order to improve their properties, grouting is a special technique developed in recent years with the repairing of cracks in rock. It is a procedure which involves grout injection into voids, fissures and cracks in rock, specifically to reduce permeability, to increase strength and durability (Zhou *et al.*, 2010; Anagnostopoulos *et al.*, 2011).

A granular ordinary type of cement in some times cannot be grouted into the fissures of the rock mass, when the crack size is very small (Anagnostopoulos, 2005). The cement particles were blocked under higher grouting pressure during the grouting process (Minh *et al.*, 2007). However, chemical grouting usually can solve this problem. Epoxy resins are widely used in chemical grouting of rock due to their advantageous properties of adhesion, good toughness and superior mechanical properties (Rogers and Daniels, 2002; Hasanpour and Choupani, 2009).

Mineral phase quantification based on powder X-Rays Diffraction (XRD) has always been an attractive option (Trimby and Prior, 1999), as it provides reproducible and representative results. The scanning electron microscope (SEM) is a powerful tool for micro

structural analysis of epoxy-repaired rock. At the Jinping Hydropower Station, epoxy resins are used to repairing the shear zone and faults, to reduce the permeability and increase strength of rock in fault. The mineral proportions and micro structural properties of epoxy-repaired rock are very important to determine the improvement of mechanical characteristics by epoxy resins. The objective of this study is a dopt electronic methods (X-ray diffraction and scanning electron microscopy) to analysis the mineral compositions and micro structural of epoxy-repaired rock.

## EXPERIMENTAL

In this section, the materials for X-ray diffraction and scanning electron microscope experiments are retrieved from the fault zone after chemical grouting. Rock should be pressed into powder for XRD analysis and cut and polished into thin slice for SEM analysis. Finally, a brief introduction for XRD and SEM methods are made.

**Materials:** A two component epoxy resin was selected for optimization and several modifications were made to the original composition provided. Figure 1 shows a polarizing microscope photo of the epoxy resin for chemical grouting of faults.

Component A is mixture of epoxy resins, component B is amine-hardener, the gravity ratio of

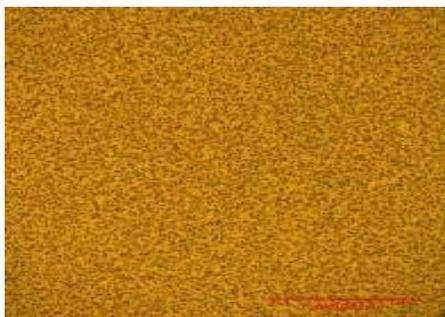


Fig. 1: Polarizing microscope photo of the epoxy resin



Fig. 2: Epoxy-repaired rocks



Fig. 3: Sample preparation for XRD and SEM tests

component A and B is 6:1. The final epoxy resin composition adequately fulfilled the project requirements and is now commercially available.

There three types of rock in the fault zone are weak weathered green schist (W), strongly weathered green schist (S) and carbonaceous green schist (C). At the beginning of field chemical grouting process, the physical and mechanical characteristics of epoxy resin should be determined. Uniaxial compressive stress of epoxy resin at the age of 28 day is about 90 MPa, a long enough coagulation time is needed for the construction process.

After the epoxy resin is completely solidified, the chemical grouting process for rock fault is finished. The epoxy-repaired rock is prepared by borehole drilling. Figure 2 shows the epoxy-repaired rocks in the fault zone.

As shown in Fig. 2, the left one is epoxy-repaired weak weathered green schist, the middle one is epoxy-repaired strongly weathered green schist and the right one is epoxy-repaired carbonaceous green schist.



Fig. 4: X-ray diffraction test system



Fig. 5: Scanning electron microscopy test system

**Sample preparation:** The epoxy-repaired rock should be pressed into powder for XRD test, the particle diameter is less than 0.001 mm. But the epoxy-repaired rock should be cut and polished into thin slice for SEM test, the length and width are all 1.0 cm, the height should be not larger than the length.

Figure 3 shows the sample preparation for XRD and SEM tests. As shown in Fig. 3, three types of rock powder are corresponding to the different rock type. The left, middle and right ones are rock powder of epoxy-repaired weak weathered green schist, strongly weathered green schist and carbonaceous green schist. There is an obvious difference in rock powder between each rock type. At the lower of Fig. 3, these specimens are thin slice of epoxy-repaired rock for SEM tests. Here we can see the epoxy resin existed in the fractured rocks.

**X-ray diffraction method:** X-ray diffraction (XRD) is an analysis method that uses X-rays to determine the range of physical and chemical characteristics of materials (Ruan and Ward, 2002; Hestnes and Soensen, 2012). This includes analysis for the types and quantities of different phases in the sample. During the semi-quantitative XRD method, the mineral proportions received by sample analysis are compared to known intensity and information found in a database distributed by the International Centre for Diffraction Data.

Representative portions of each epoxy-repaired rock were more finely ground in a ring-grinder mill and the particle diameter is less than 0.001 mm. The minerals in each case were identified from the diffractograms by reference to the rock Powder Diffraction File. Figure 4 shows the test system for X-ray diffraction.

Quantitative analyses of the minerals present in each sample were made using commercial software based on the full-profile XRD analysis technique. If the internal standard weight percentage is X, will be greater than the weighted amount Y. The mineral proportion in the tested sample is given by the following equation:

$$W_a = 1 - (Y/X) \tag{1}$$

where,

- W<sub>a</sub> = The mineral proportion in the tested sample
- X = The mineral amount found by the XRD program
- Y = The weighed amount of powder sample.

**Scanning electron microscopy** Scanning electron microscopy analyses on extracted micro structural characteristics of epoxy-repaired rock provide the detail morphology and texture of such grains. This is important since anthropogenic magnetic particles can be distinguished from specific morphology. Figure 5 shows the test system for scanning electron microscopy.

Selection criteria for grain analyses by SEM were based on the subjective differences observed in the morphology and texture behaviors. The filling situation of epoxy resins in the fractured rock can be determined by SEM method.

## RESULTS AND DISCUSSION

In this section, some test results about the mineral proportions and micro structural characteristics of epoxy-repaired rock are illustrated.

**General:** Because of there are lots of cracks and voids existed in the original rock at fault zone, so that the porosity of rock is very large. After the chemical grouting process, the fractured rock is repaired by epoxy resins and then the porosity of rock will be decreased. Table 1 shows the variation of the rock porosity before and after chemical grouting.

As shown in Table 1, the porosity of original fractured rock is about 15%-20%, after the chemical grouting by epoxy resins, some of the cracks and voids are repaired by epoxy particles, resulted in the decreasing rock porosity. Its mean that, the quality of fractured rock is improved by the epoxy resins. Figure 6 shows a polarizing microscope photo of the epoxy-repaired rock sample.

As shown in Fig. 6 (compared with Fig. 1), the cracks and voids in the fractured rock are filled with epoxy resins.

**Mineral compositions:** Several test for mineral compositions of epoxy-repaired rock by X-ray diffraction method are carried out. Figure 7 shows the X-ray diffraction analysis results for mineral proportions of epoxy-repaired rock.

Table 1: Variation of the porosity before and after grouting

Rock type	Original (%)	Epoxy-repaired (%)
W	14.73	9.68
S	18.32	12.16
C	20.5	13.25

Table 2: Mineral compositions of the epoxy-repaired rocks

Rock type	W	S	C
SiO <sub>2</sub>	37.63	40.46	54.32
CaCO <sub>3</sub>	30.56	28.28	16.13
FeO (OH)	12.65	10.86	6.56
Al <sub>2</sub> O <sub>3</sub>	5.64	4.86	6.86
Proportion (%)	3.29	3.58	2.65
K <sub>2</sub> O	2.35	2.65	3.21
FeO	2.16	3.16	1.67
Na <sub>2</sub> O	1.15	1.57	2.31
Epoxy	3.45	4.36	5.61

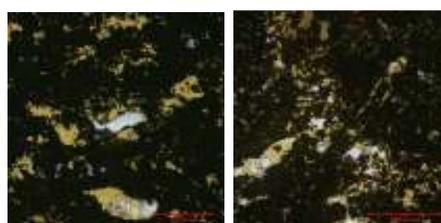
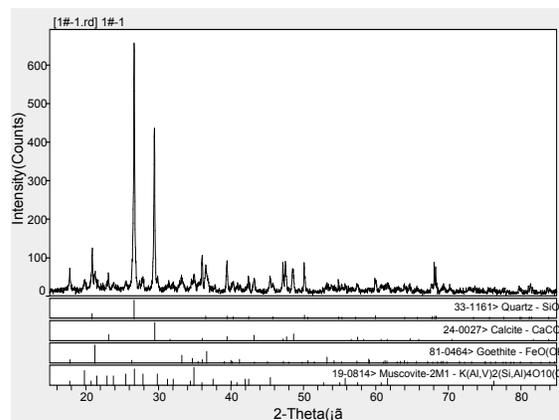
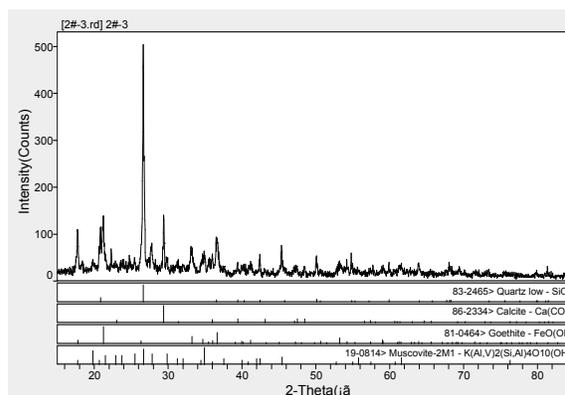


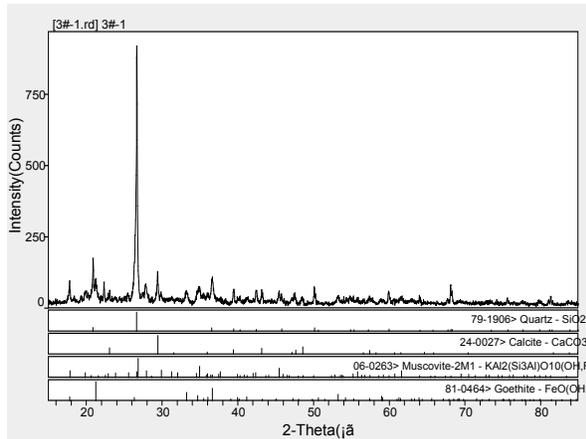
Fig. 6: Polarizing microscope photo of the epoxy-repaired rock sample



(a)



(b)



(c)

Fig. 7: X-ray diffraction analysis results: (a) weak weathered greenschist; (b) strong weathered greenschist; (c) carbonaceous greenschist

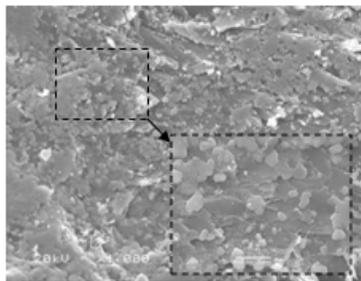


Fig. 8: Microstructural characteristics of the epoxy-repaired rock

As shown in Fig. 7, there is an obvious difference between each mineral particle, so that the mineral proportions of epoxy-repaired rock can be determined by XRD method. Table 2 shows the mineral compositions of the epoxy-repaired rocks.

As shown in Table 2, the proportion of  $\text{SiO}_2$  is the largest one of mineral, about 40%-50%. The proportion of epoxy resins is about 3.5%-5.5%, compared with Table 1, after chemical grouting, the cracks and voids in the fractured rock are partly repaired. Some smaller cracks and voids can not be filled by epoxy resin particles.

**Microstructural characteristics:** The micro structural characteristics of epoxy-repaired rock are analysis by the SEM method. Figure 8 shows the typical micro structural characteristics of the epoxy-repaired rock.

As shown in Fig. 8, there is lots of epoxy resin particles existed at the crack surface, showing a discrete distribution and can not connect with each other. That mean the filling of epoxy resin in the cracks and voids is partly.

## CONCLUSION

Fractured rock can be repaired by chemical grouting of epoxy resins, how to determine the improvement of epoxy resin for fractured rock is very important. In this study, XRD and SEM methods are adopted to make certain the chemical grouting effect on rocks. Test results show that, the cracks and voids in rock are partly filled by epoxy resins; the proportion of epoxy resin in rock is about 4.5%.

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