LOS ALAMOS NATIONAL LABORATORY

YUCCA MOUNTAIN PROJECT CHANGE REQUEST

CHANGE REQUEST No. 118

DATE 1/12/90

NO.

COPY

PROCEDURE No. TWS-ESS-DP-124, R0 Use of Binocular Microscope in Fracture

CHANGE REQUESTED:

Section 5.6: Change title of section to "Accept/Reject Criteria"

Add sentence to end of paragraph:

"Photographs and photographic slides taken as described in Sections 5.2 and 5.3 of this procedure will be accepted if they show the features that were photogaphed."

REASON FOR CHANGE:

To provide a clearly identifiable accept/reject criteria section.

CHANGE REQUESTED BY Barbara Carlo DATE 1-17-90

REVIEWED BY Stephen J Bolivar DATE 1-17-90

QAPL APPROVAL D P Jones DATE 1/25/90

TPO APPROVAL R J Herbst DATE 1/25/90

EFFECTIVE DATE January 25, 1990

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NO. TWS-ESS-DP-124, R0

USE OF BINOCULAR MICROSCOPE IN FRACTURE MINERALOGY STUDIES

Effective Date 2/25/89

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2-10-89
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USE OF BINOCULAR MICROSCOPE IN FRACTURE MINERALOGY STUDIES

1.0 PURPOSE

The purpose of this procedure is to describe the use of the binocular microscope in examining fracture-lining minerals and preparing samples for examination and analysis using other methods to determine their mineralogy and paragenetic relationships.

2.0 SCOPE

The procedure will be used in studying fracture coatings in drill core, shaft, drift, and outcrop samples for the Yucca Mountain Project, and may be used in examination of other samples when appropriate.

3.0 REFERENCES

This procedure is written to conform to LANL-NNWSI-QAPP R2 and TWS-QAS-QP-05.2,R1. LANL procedures referenced are:

TWS-QAS-QP-03, Document Control Procedure
TWS-QAS-QP-13.1, Handling, Storage, and Shipping Procedure
TWS-ESS-DP-101, Sample Identification and Control for Mineralogy-Petrology Studies.

YMP procedures referenced are:

AP-6.3Q, Use of Sample Management Facility by Yucca Mountain Project Participants
AP-SOC-1, Approval Procedure of Request for Yucca Mountain Project Geologic Samples
BTP-SMF-005, Examination of Yucca Mountain Project Samples by Participants
BTP-SMF-006, Removal of Whole Core and Other Specimens from Yucca Mountain Project Samples for Shipment and Remnant Return

4.0 RESPONSIBILITIES

The PI has the responsibility of organizing and overseeing all operations. The PI may assign appropriate tasks to trained personnel. Investigators performing any part of this procedure will keep a written record in their own laboratory notebooks of what has been done. It is the responsibility of users of this DP to adhere to the procedure. Any deviations from the procedure require approval of the PI and will be documented in the laboratory notebook.

5.0 PROCEDURE

5.1 Operating conditions

No special environmental conditions other than those present in the normal laboratory or office are necessary for this procedure.

5.2 Photodocumentation of samples

The original sample is photographed to record the the appearance of the whole piece of core or rock sample. Photographs are taken of every fracture surface and trace showing both the amount of coverage on open fracture faces and the orientation of the fracture with respect to the core cylinder and any visible lineations in the rock. Photographic slides taken using Tungsten 160 film work well for this, but may not be the only choice. Slides are stored in slide preserver pages in labeled ring binders. The sample is not broken or cut until the slides are inspected for adequacy (focus, identifiable features).

5.3 Microscopic examination of samples

Samples are examined under the binocular microscope, typically at 6-100x magnification. Any model of binocular microscope capable of 50x magnification is acceptable. Some samples require washing with water before fracture minerals can be studied. Care is taken during washing to preserve the fracture coating minerals while rinsing away the accumulated detritus from coring or sampling. Both closed and open fractures are examined and described in terms of orientation (as well as can be determined), and coating. Descriptions of fracture coatings including visual estimates of percent coverage of the open fracture face, color of coating(s), inter-relationship of different coatings, morphology of crystals or patches of microcrystalline material, and hardness relative to other coatings and the rock matrix are written in the investigator's laboratory notebook. Photographs or slides are taken of representative and/or significant features of the fractures through the binocular microscope, and sample number and magnification are recorded. (Tungsten 160 slide film reproduces the color of the sample without requiring a daylight filter on the tungsten light source.) When the photos (slides) are returned from processing, they are labelled with sample number and magnification. Slides are stored in slide preserver pages in labeled ring binders. No features of the fracture coating are destroyed or changed by sampling until the photos have been examined for clarity.

5.4 Preparation of SEM sample

A representative fragment of the open fracture is broken off the original sample for scanning electron microscope (SEM) examination. Optimum size is about 1 cm sq, but samples may be up to about 3x2 cm or as small as 1/2 cm on a side. Thickness of the sample is not more than 2 cm. If the surface opposite the fracture coating is not flat, the SEM sample is abraded until the sample will sit with the fracture surface up. An aluminum SEM stub is labeled with the sample number (and fracture number or letter if assigned). The SEM sample is then glued to the aluminum stub with water soluble glue. When this has dried, silver paint is traced across the glue from the exposed aluminum to the fracture surface, trying not to get any silver on the fracture coating. (It becomes obvious in the SEM if there is silver on the surface, but it obliterates features on the sample). A color Polaroid photo is taken of the mounted SEM sample to aid in location of features once the sample is gold or carbon coated. The sample is then stored in a SEM sample storage box that has been labeled with the drill hole number.

5.5 Preparation of XRD sample

After the binocular microscope pictures have been filed and the SEM sample taken, the fracture surface is scraped using a tungsten carbide scraper. Care is taken to avoid contamination with rock matrix. Observations on hardness, difficulty of scraping, and ease of separating coating from matrix are made in the notebook. An area of at least 1 cm sq. is left unscraped on the

original fracture surface for future reference. The coating scraped off the fracture surface is handpicked to remove as much as possible of the matrix fragments contaminating the sample. An estimate of the remaining matrix contamination, based on differences in hardness, texture and color between the coating and matrix, is written in the notebook. This entire process is performed using the binocular microscope. A small vial is labeled with the sample number, and the scraped XRD sample is stored in this vial. Because of the small amount of sample usually available from fracture surfaces, the XRD powders cannot be ground using the Brinkman Grinder, but must be powdered by hand using a small mortar and pestle.

5.6 Data generated during performance of this Procedure

Data generated during performance of this procedure are descriptions of fracture coatings and of sample splits taken of those coatings. Descriptions are entered in the investigator's laboratory notebook and acceptance of the data is determined by review of that notebook in accordance with TWS-QAS-QP-03. No methods of data reduction are used in this procedure.

6.0 QUALITY ASSURANCE REQUIREMENTS

6.1 Sample shipping and handling

Samples will be obtained through the SMF following YMP procedures AP-6.3Q, AP-SOC-1, BTP-SMF-005 and BTP-SMF-006 as applicable. Once samples are received at Los Alamos, they are handled according to TWS-QAS-QP-13.1 and TWS-ESS-DP-101. Samples already under curation at Los Alamos will also be studied using this procedure. Samples must be shipped and stored in a manner to preserve the fracture coatings intact.

6.2 Records

Records generated by this activity include the following:

a. Photos (slides, Polaroids, negatives and prints) stored in the office of the PI.
b. Notebooks, stored in office of the investigator making the entries, reviewed and duplicated following TWS-QAS-QP-03, then tranmitted to the record processing center when they are no longer needed for reference.

6.3 Training

The preparer of this procedure is considered trained to implement the procedure and to train others in its use. Training for other investigators will include reading this procedure and may requre additional instruction by the PI, depending on the background and experience of the investigator. Competence in the method is documented in the personnel certification file.

6.4 Calibration

No calibration is required for methods described in this procedure.

6.5 Procedural deviations

Procedural deviations are documented in the notebook of the investigator together with an evaluation of the effects of the deviations on the results.