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### Title

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**Visualization of microcrack anisotropy in granite affected by a fault  
zone, using confocal laser scanning microscope**

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## **Abstract**

Brittle deformation in granite can generate a fracture system with different patterns. Detailed fracture analyses at both macroscopic and microscopic scales, together with physical property data from a drill-core, are used to classify the effects of reverse fault deformation in four domains: (1) undeformed granite, (2) fractured granite with cataclastic seams, (3) fractured granite from the damage zone, and (4) foliated cataclasite from the core of the fault. Intact samples from two orthogonal directions, horizontal (H) and vertical (V), from the four domains indicate a developing fracture anisotropy toward the fault, which is highly developed in the damage zone. As a specific illustration of this phenomenon, resin impregnation, using a confocal laser scanning microscope (CLSM) technique is applied to visualize the fracture anisotropy developed in the Toki Granite, Japan. As a result, microcrack networks have been observed to develop in H sections and elongate open cracks in V sections, suggesting that flow pathways can be determined by deformation.

The complex interplay of deformation and fluid flow along microcracks developed in granite, and the potential of CLSM is inferred as an important tool for understanding the relationship between brittle deformation and fluid flow are discussed.

Key words: fracture anisotropy, granite, confocal laser scanning microscope, and physical properties

## **1. Introduction**

Transport properties of rocks are important for the evaluation of subsurface contamination and safety management. At shallow crustal levels, faults and fractures characterize the main deformation and are the main factors in increasing flow paths and controlling the migration of contaminant materials in crystalline rocks. In fault zones, where brittle deformation dominates, the connectivity of microcracks is one of the main factors controlling hydraulic properties. Contaminants such as radionuclides may diffuse into pore structures, increasing the mass transport travel time and the retardation potential of a geological system. Knowledge of granite pore features such as connectivity, tortuosity, effective porosity and permeability - from intact to highly fractured fault rocks - are important input data in models of groundwater systems used for mass transport and performance assessment. An understanding of pore space requires knowledge of fracture network spatial distribution. Several studies have addressed the physical properties of crystalline rocks, focusing in sorption (Moreno et al., 1997), diffusion into rock matrix (Neretnieks, 1980) and migration pathways (Frieg et al., 1998; Hellmuth et al., 1993; Yoshida et al., 2000).

To characterize fracture systems at different scales is difficult, because of their complexity, the heterogeneity of fracture networks, stress dependence, and spatial and temporal relationships with major geologic structures. Fracture geometry can be characterized from one or two-dimensional observations at macroscopic and microscopic scales. Previous methods for obtaining three-dimensional fracture images usually involved impregnation and sample sectioning and exposure, either using autoradiography,

CCD image technique, or scanning electron microscope (SEM) prior stacking (Hellmuth et al., 1993, 1999, and Frieg et al., 1998). Other approaches to obtain full high-resolution characterization of pore structure used x-ray computerized tomography (Montemagno and Pyrak-Nolte, 1995; Cislárová and Votrubová, 2002; Coker et al., 1996; Farber et al., 2003; Wildenschild et al., 2002) and laser scanning confocal microscopy (CLSM) (Fredrich et al., 1995, Montoto et al., 1995, Shimizu and Shimada, 2002; Onishi and Shimizu, 2003). These methods provide high-resolution, nondestructive pore analysis of porous as well as fractured media. In this study, we characterize and evaluate the spatial distribution of microcrack networks related to faulting, using an improved method of the resin impregnation technique combined with CLSM.

## **2. Borehole Samples**

The Toki Granite of Upper Cretaceous age (c.a. 70 Ma) intrudes into the Mesozoic sedimentary basins in the Tono region, Southwest Japan (Figure 1). The granite body is composed of medium- to coarse-grained biotite-granite and biotite monzogranite, which are in turn composed mainly of plagioclase, K-feldspar, quartz, biotite, and a small amount of accessory minerals such as zircon, apatite, ilmenite, sericite, muscovite, epidote, and calcite. The main geologic structure in the Tono region is the Tsukiyoshi Fault. It is a relatively young (late-Tertiary), reverse E-W striking fault that dips about 70° to the south. An overall displacement of 30 m is estimated from the overlying sedimentary rocks (Toyokura et al., 2000) (Figure 2). The Tsukiyoshi Fault is a case study for site characterization and performance assessment for the Japan Nuclear Cycle Development Institute – JNC.

Samples were taken from the borehole MIU3, a 1000 m deep MIU3 borehole. The top of the granite is found at 91.4 m depth and the center of the fault zone center at 707 m (Toyokura et al., 2000) (Figure 3).

To constrain the relationship between microcracks and major structures, we selected core samples from four distinct domains: (1) fresh undeformed granite, (2) granites with fractures sealed by chlorite, (3) fractured granite from the damage zone and (4) foliated cataclasite from the center of the fault zone (Figures 3, 4). Half of the core was used for petrographic analysis and characterization of microcracks by CLSM, the other half, for measuring porosity, pore size distribution, and permeability.

As shown in Figure 3, most fracture planes observed in the core are subvertical. Horizontal disk fractures induced during core retrieval and stress release are also observed. For this study, we analyzed samples in two orthogonal sections with respect to the main subvertical fracture orientation. Sections taken parallel to the main vertical fractures are labeled as V samples, while sections taken normal to them are labeled as H samples.

### **3. Petrographic Analysis**

The typical samples from the four distinct domains mentioned above can be described as follows:

(1) The undeformed granite is medium-grained biotite granite with a grain size ranging from 0.2 to 1.5 mm, fresh in hand specimen with no visible fractures (Figure 3a). Under the optical microscope, microcracks are weakly developed in the fresh granite, with most of them observed in quartz grains. As identified by x-ray diffractometry XRD

(Table 1), weak fine clay mineral alteration is found in the core of the plagioclases (Figure 4a, 4b). Overall, the granite seems clear and fresh in both H and V sections, with no major change in texture or structure.

(2) Samples with fractures sealed by chlorite are taken from a domain of medium- to coarse-grained granite, with grain sizes ranging from 0.5 to up to 5 mm (Figure 3b). The sealed fractures, referred to as cataclastic seams in this study, have variable width, reaching several millimeters. Under the microscope, most of the seams are filled with fragmented minerals and fine chlorite (a finding confirmed by XRD analysis). In general, mineral alteration is weak and mostly concentrated at the core of the plagioclases. Microcrack anisotropy is not clear when the cataclastic seams are well developed (Figure 4c and 4d).

(3) The domain of the damage zone includes medium- to coarse-grained granite, with grain sizes ranging from 0.5 to 5 mm (Figure 3c). This domain is characterized by pronounced fracturing observed at both macroscopic and microscopic scales, and includes gouges and breccias not larger than 20 cm wide. The most prominent evidence of fracture anisotropy is observed in the damage zone. In this zone, microcracks have developed, identified by a clear network of microcracks and open fractures on H and V sections, respectively (Figure 4e, 4f). This pattern is consistently observed in all samples from the damage zone. In addition, mineral alteration is visible in the core of the plagioclases and in biotite cleavages.

(4) As shown in Figures 3d and 4g the core of the fault zone is composed of foliated cataclasite. In hand specimen, the shear zone is characterized by overall fragmentation and mineral size reduction. Grain size is highly variable, ranging from less

than 0.1 to 1.5 mm. Under the microscope a foliation in the cataclasite is defined by sheared biotite, broken quartz, and feldspars (Figure 4g). XRD data indicate sericite, halloysite, chlorite, and fine calcite as matrix-filling minerals in the core of the foliated cataclasite, possibly the result of water-rock reactions. Calcite is concentrated adjacent to the fault core in the shear zone. This zone is characterized by very fine minerals, usually smectite, sericite, and chlorite, surrounding other small grains such as quartz and feldspars.

#### **4. Porosity Measurements**

In this study, porosity measurement was carried out using a helium pycnometer and by mercury-injection porosimetry. The former gives the bulk porosity, using compressible helium gas. It is more efficient for low-porosity rocks, but does not give pore-size distributions. Mercury-injection method allows to estimate porosity and the pore-size distribution by measuring the volume of mercury injected in dry cubic samples. It requires external pressure to force the liquid mercury into the pore spaces. Pore size is estimated by the volume intruded into the pores as a function of the applied injection pressure. The maximum pressure used in this study was 400 MPa, and the maximum volume of mercury intruded was  $0.003 \text{ mLg}^{-1}$  for most samples and  $0.02 \text{ mLg}^{-1}$  for the fault zone rock. Pore size ranged from 0.001 to 100 microns.

For the fresh granite and granite with cataclastic seams the porosities measured by the helium pycnometer are slightly higher than those obtained by mercury injection (Table 2 and Figure 5). The variation in pore diameter with depth is shown in Figure 6. The undeformed granite shows higher pore diameter than the cataclastic seams, probably

because of presence of chlorite along the fractures (as detected by XRD). In the damage zone, the pore diameter varies more and increases toward the fault zone. The high pore diameter in the core of the fault zone may be related to rock fragmentation and shearing.

A plot of representative samples from each domain shows the variations in pore diameter (Figure 7). The pore-size diameter changes from bimodal to multimodal toward the fault zone. It is likely that fault-related fracturing might have altered the pore structure, generating microcracks with different pore size. In addition, in foliated cataclasite, the volume of mercury is one order of magnitude higher in the fault sample than in samples from other domains, reflecting the effects of intense deformation and grain-size reduction in the fault zone. The variation in pore diameter with porosity for representative samples from each domain is illustrated in Figure 8.

## **5. Characterization of Microcracks under Laser Scanning Microscope**

The use of a confocal laser scanning microscope (CLSM) allows visualization of pore structure in samples impregnated with fluorescent resin (Fredrich et al., 1995, Montoto et al., 1995). Applied the technique to the Toki granite, CLSM enabled us to visualize the fracture network in two and three dimensions and infer the microcrack networks, their connectivity, and geometry.

### **5.1. Apparatus**

The CLSM used in this study is a laser scanning microscope FLUOVIEW (OLYMPUS Co.) equipped with a K-Ar laser source (wavelengths of 488 nm, 568 nm, and 647 nm). We captured digital intensity maps of fluorescent light excited by blue (488

nm) laser beams with the fluorescent intensity represented by 12-bits (i.e., 4,096 gray level) digital images. The optical resolution of the CLSM depends upon the numerical aperture (NA) of the microscope. All digital images shown in this study are displayed using an objective lens with a magnitude of ten (Uplan Apo 10xNA = 0.3). As a result, for a digital image size of 1024 x 1024 pixels, the optical resolution is 0.75 microns in lateral (x-y) sections and 6.94 microns in the z direction.

## **5.2. Resin Impregnation**

The impregnation method was performed using low-viscosity resin (less than 1.5 mPas) with a fluorescent dye in coupon samples a size of 40 x 25 x 25 mm<sup>3</sup> at room temperature. Samples were pre-dried for 48 hours at a temperature of 85–90°C. The resin used was methylmetacrylate pre-mixed with fluorescent dye. The resin was injected under vacuum (76 psi) and left until degassed, indicating total impregnation of the rock matrix. The samples were then heated to 80°C in a water bath. After hardening, the samples were cut at the center and polished to make thin sections. However, the impregnated thin sections were made thicker (>150 µm) than those used for standard petrographic analysis, because optical slicing under CLSM to produce a 3-D images requires thicker sections (Fredrich et al., 1995; Montoto et al., 1995; Onishi and Shimizu, 2003).

The impregnated coupons were attached to a microscope stage that could move them laterally. Each scan of the selected area produced a digital image of 1024 x 1024 pixels covering 1,414 square-micron meters. To construct an image, we scanned ten slices, using 2 µm vertical steps, stacked and normalized to maximum intensity. This

procedure was followed to avoid intensity variations caused by surface irregularities. Profiles taken along the x-z direction were scanned up to 200 to 250  $\mu\text{m}$ , using a 0.5  $\mu\text{m}$  step size.

### **5.3. Image Processing**

Image processing was applied to two-dimensional digital images in the x-y and x-z planes. The x-y plane gave the distribution of microcracks, and its distribution and the x-z plane showed the fracture geometry as the resin filled the microcracks. A commercial software (Image Pro Plus, Media Cybernetics) was used to improve image quality by contrast manipulation and application of spatial filters (Gonzalez and Woods, 2001). Gaussian and median filters were applied to remove artifacts and smooth the images. Resin intensity in the images acquired by CLSM can be greatly affected by the use of the filters during image processing. As a result, significant variations on fracture porosity and aperture might be expected. For low porosity rocks, such as granite, these effects increase inaccuracy, and for this reason we did not use digital images for the quantitative analysis.

Digital images were used to collect qualitative information on the x-y plane and x-z plane cross sections for samples in both (H and V) directions, as shown in Figure 9. For quantitative purposes, unprocessed data could be used to obtain aperture information; extensive work on the use of algorithms to quantify porosity from digital images has been conducted in porous media (Coker and Torquato, 1995; Fredrich and Lindquist, 1997; Lindquist et al., 1996; Lindquist et al., 2000). Although the filter in the CLSM blocked most of the noise, the threshold between pore space and matrix was not well defined for

tiny cracks in granite. Additional work is under way to optimize the aperture information in granite under CLSM. Better resolution can be obtained using lenses with high magnification that focus on single fractures; however, it is difficult to characterize the entire fracture network. By increasing lens magnification the pixel resolution is increased, but the covered area is reduced. Our purpose is not to study a single fracture but to understand the relationship between microcrack patterns resulted from deformation.

#### **5.4. Characterization of Microcracks by a Laser Scanning Microscope**

A laser beam is able to detect the fluorescence from the resin through grains and open cracks up to a depth of 250 microns. However, the laser usually attenuates with depth, either by light refraction or reflection from silicates and because of crack geometry. That is, the fluorescent decreases in intensity with depth.

The pore size distribution from mercury porosimetry shown in Figure 7 has modes of 0.1, 1, and 10 microns. Because of the 0.75 micron resolution of the CLSM, pores of size 0.1 micron were not observed. In undeformed granite (Figure 9A), the H and V section digital images show, indistinctly, that the resin fills most of the grain boundaries and intragranular fractures. Mineral grains are usually dark under the fluorescence mode of CLSM, however, the fluorescence surrounding the grains indicates that the pore space has been filled with the resin. Resin impregnation in both directions is very similar and is concentrated along grain boundaries and intragranular fractures. Note that fluorescence intensity along pore spaces shows large variations in the x-y plane. A similar result is observe in the x-z profiles taken along profiles a and b of Figure 9A, where the resin is observed bending with depth. The images also show that the intensity

attenuates as the laser travels through the sample. Differences in mineral composition can affect laser attenuation, but the x-z images clearly show the pore geometry with the resin filling the grain boundaries.

As a result, the width of fluorescent intensity observed in the x-y plane is wider for non-vertical microcracks, because it includes the interference of intensity of inclined structures resulting in an apparent thickness. Hence, the quantification of aperture under CLSM in two-dimensions is greatly influenced by laser reflection and/or refraction with depth.

Similarly, in cataclastic seam domains, the resin is concentrated along the wall of the seams, as observed in the H section and along microcracks related to grain boundaries and intragranular fractures in both H and V sections (Figure 9B). Profiles a and b, observed in the H sections of the cataclastic seams, show that the seams have a limited depth as the laser attenuates through the sample. Likewise, in the V section, laser attenuates with depth along the grain boundaries. In addition, laser intensity noticed along the seams is slightly high, rather than along the grain boundaries.

In the damage zone, the two directions indistinctly show different microcrack distribution features under CLSM (Figure 9C). As deformation increases toward the fault zone, microcracks anisotropy is very clear. In general, the resin is concentrated along network microcracks in the H sections, with the resin filling grain boundaries, and intergranular and intragranular fractures. Conversely, the resin fills more efficiently along subparallel open cracks in the V sections. As observed in Figure 9C, the V sections show open cracks cutting thorough minerals as parallel fracture sets.

In the sample from the fault zone, no microcrack anisotropy is observed, due to overall rock fragmentation and grain size reduction. The resin is highly concentrated in the matrix surrounding mineral fragments (Figure 9D). The shallow presence of resin in the profile may indicate resin attenuation in the matrix, possibly caused by a high concentration of fine clay minerals (as determined by XRD analysis).

## **6. Discussion**

This study is an attempt to examine the overall deformation in granite caused by a reverse fault, through the visualization of the microcracks associated with the fault. The focus is on the microcracks anisotropy developed in the hanging wall of the Tsukiyoshi Fault. Detailed examination of fractures in drill core samples, using petrographic, resin impregnation, and petrophysical (porosity and pore-size distribution) measurement, indicates that the geometry of microcracks is affected by deformation and therefore can affect flow properties of the rock mass.

Petrographic observation indicates the development of microcrack anisotropy toward the fault zone, which is highly concentrated in the damage zone. In a vertical borehole, microcrack anisotropy is observed in both orthogonal directions. Increasing development of microcrack networks in the H sections and along open cracks in the V sections are both clearly identified in the Toki granite. Although the microcracks may originate from granite emplacement, caused by uplifting and hydraulic processes, the indistinctly feature observed toward the fault zone indicates the effect of the Tsukiyoshi Fault on the development of microcracks. In a conceptual model of a fault zone, Caine et al., 1996 classified a fault zone in terms of damage zone distribution. In this case, it is

important to understand how deformation affects the flow patterns along each fault zone domain by analyzing the fracture geometry. In addition, the type of lithology, the response to the stress field, and the geological history of the fault are important components to be evaluated.

The result of our detailed macro- and microscopic study of fracture network characterization and of the physical properties of granite indicate that deformation may influence the flow process.

Bulk porosity increases toward the core of the fault zone, with the highest porosity observed in the foliated cataclasite in the fault zone itself (as shown in Figure 5). The values of porosity in the Toki Granite are similar to other granitic rocks reported so far (Brace et al., 1968; Kiyama et al., 1996; Morrow and Locker, 1997; Yoshida et al., 2000). The relationship between porosity and fault zone structures is similar to that studied in other places (Caine et al., 1996; Evans et al., 1997; Geraud et al., 1995; Seront et al., 1998).

Here, a new approach for characterizing pore geometry in granite using CLSM is presented and used as a tool to identify pore space. In previous studies involving resin impregnation in crystalline rocks, pore structures are classified into grain boundary or intergranular, intragranular, and cleavage types (Fredrich et al., 1995; Frieg et al., 1998; Hellmuth et al., 1993; Montoto et al., 1995; Nishiyama and Kusuda, 1994; Ota et al., 1998; Yoshida et al., 2000). However, no clear relationship between major geologic structures is described.

The undeformed granite has usually no visible fractures either in hand-specimen samples or under the microscope. It is very unlikely that such low porosity (<1%) Toki

granite would have resin filling along its grain boundaries, intragranular fractures, and cleavage as observed under CLSM.

Additionally, the bimodal pore-size distribution observed in fresh granite confirms the existence of different types of microcracks. Because the samples are taken from the hanging wall of the fault, the network microcracks can be related to block fault movement. As a result, stress release would be responsible for creating indistinctive open cracks along grain boundary and cleavage, with no anisotropy in the microcracks.

Microcracks in a domain of cataclastic seams are similar to undeformed granite, but in addition, resin is highly concentrated along cataclastic seams. The low porosity, relatively small pore distribution, and presence of chlorite-rich seams are mostly related to crack sealing by hydrothermal processes. The anisotropy is not well developed, because the domain is not in the damage zone, and chlorite seams are unaltered.

As the deformation and alteration increases toward the fault core, the increase in porosity is directly related to fracture density. The pore geometry is more heterogeneous in the damage zone. In addition, clear network microcracks in the H sections and open cracks in the V sections are observed, resulting in the development of microcracks anisotropy. Such effects can be enhanced by stress release during core retrieval. The distinct patterns in microcracks observed in our study suggest that the damage zone is sensitive to deformation and, therefore, might promote focus flow along a set of main parallel fractures or diffuse flow along network microcracks. Most of the experiments and simulations along the fault zone describe high flow along the fault plane (Caine and Forster, 1999), but as we described, network microcrack in the damage zone also plays an important role in flow process.

In the fault rock, with bulk porosity up to 6% and variable pore diameter, pore space is highly concentrated in the matrix, as shown by resin impregnation. This is attributed to the formation of shear zones, fragmentation, grain size reduction, chemical alteration, and mineral precipitation. The complexity of deformation at the center of the fault, as well as the large amount of fine clay minerals, possibly attenuates the light from the deeper parts of the sections during CLSM analysis. This might explain why (in the profile) the resin is concentrated at a shallow depth.

More detailed studies should be performed to understand the influence of anisotropy on microcracks and fluid flow. The CLSM is useful in visualizing fractures in two and three dimensions and in providing qualitative information about fracture networks. A quantitative result combining the CLSM and permeability will be published elsewhere.

## **7. Conclusion**

Tsukiyoshi Fault is a case study for site characterization and performance assessment for the Japan Nuclear Cycle Development Institute. Investigation of deformation and fracture systems in the Toki Granite reveals that faults affects the granite properties by inducing different types of fractures both at a macroscopic and microscopic scale. Fracture anisotropy in the damage zone is related to deformation of the fault zone. Detailed investigation of fractures and microcracks indicate the influence of the fault zone along heterogeneous microcracks. The anisotropy in microcracks can be characterized by petrographic analysis and the resin impregnation method, combined with CLSM.

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**Figure captions:**

Figure 1. Location of the MIU (Mizunami Underground) Site in Gifu Prefecture, Japan. The geologic map shows the distribution of the Toki Granite and the main geologic structure in the area, the Tsukiyoshi Fault.

Figure 2. Geological block model of the MIU site showing textural variations of Toki Granite and the Tsukiyoshi Fault.

Figure 3. MIU-3 borehole stratigraphic column. The Tsukiyoshi Fault is located at 707 m depth. (A) Fresh and undeformed granite is from 350 m depth. (B) Fractured granite with cataclastic seams is from 575 m depth. (C) Fractured granite from damage zone at 698 m depth. (D) Foliated cataclasite from the core of the fault.

Figure 4. Photomicrograph taken in cross-polarized light of four domains in the Toki Granite. The left side of the photomicrograph displayed is taken from a horizontal (H) section, and on the right side from a vertical (V) section. Note that no visible fractures are observed in the minerals within the fresh undeformed granite (A and B). In the domain of cataclastic seams (C and D), fracture-filled seams cut through minerals with some small displacement in the plagioclases. Anisotropy in microcracks are not well developed, as observed in the damage zone domain (E and F), where clear network microcrack in the H section and parallel open fractures in the V section are evident. The foliated cataclasite from the core of the fault zone shows overall shearing (G). A schematic illustration of the core is shown on the left.

Figure 5. Variations in bulk porosity with depth, determined using two methods: mercury injection and helium pycnometers. Core of fault zone at 707 m depth.

Figure 6. Variations in pore diameter with depth. Note an increase in pore diameter in the damage zone (at 575 m depth), and toward the core of the fault zone (at 707 m depth).

Figure 7. Equivalent pore diameter from the four domains showing the variation in pore distribution.

Figure 8. Cumulative volume curves illustrating the relationship between pore diameter and porosity using representative samples from the four domains.

Figure 9. Digital images from a laser scanning microscope of representative samples from four domains show microcracks filled by resin. . The images are 2-D in the X-Y (two images) and in the X-Z planes. Each image has 1024 x 1024 and 1024 x 145pixels, respectively, and the intensity level ranges from 0 to 4096 gray levels. In the undeformed biotite granite, both H and V sections show resin impregnation, mainly along grain boundaries and some intergranular fractures. The X-Z profiles indicate the connectivity of the fractures with depth (Figure 9A). A similar result is achieved in the domain of cataclastic seams, with resin also filling intragranular fractures from the filling minerals (Figure 9B). Anisotropy is clearly observed in the damage zone, with network microcracks developing in the H sections and parallel open fractures in the V sections.

The same is observed in the X-Z profiles (Figure 9C). High but shallow concentration of resin is observed in the core of the fault (Figure 9D). See text for details.

### **Tables**

Table 1. Results of X-ray diffractometry (XRD) of fault-related rocks

Table 2. Summary of porosity and pore size distribution of samples from the MIU-3 borehole