Size effect and correlation with hardness of the microscopic fracture toughness of the minerals of granite

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ABSTRACT: Microscopic fracture toughness (MFT) tests on the minerals composing the Inada granite (quartz, K-feldspar, plagioclase, biotite) were conducted to elucidate the fracture phenomenon of rock. MFT tests were conducted with three cantilevered specimens of micrometer size. The MFT of mineral was in the order of quartz, plagioclase, K-feldspar, and biotite from the largest value. Those MFT were lesser than the macroscopic fracture toughness of the Inada granite evaluated by the SCB test. In addition, the mineral grain boundary MFT was smaller than the mineral MFT. We found no significant scale dependence for the MFT itself. It was suggested that macroscopic fracture toughness of Inada granite may be controlled by quartz, which has a large composition ratio and large MFT. Furthermore, it was clarified that the MFT of each mineral has a positive correlation with the Vickers hardness, which are indicators of the hardness of the minerals.

Keywords: Microscopic fracture toughness, Mineral grain, Mineral grain boundary, Size effect, Hardness.

1 INTRODUCTION

In relation to the effective use of supercritical geothermal resources and the geological disposal of high-level radioactive waste, what is one of the essential to achieve them is "understanding and control of rock fracture". Rocks are heterogeneous bodies composed of rock matrix and various mineral particles. Fracture planes in rocks are known to occur within mineral grains and through grain boundaries (Kataoka et al., 2018). Such fracture is peculiar to rock as a heterogeneous body. It is considered that rock fracture is largely caused by differences in fracture toughness and deformation properties of rock matrix, mineral grains, and grain boundaries. In addition, it is thought that their fracture toughness and deformation properties are also affected by temperature, pressure, and time axes. Therefore, we should evaluate these factors more accurately before discussing failure.

In this study, we measure the fracture toughness of rocks, their constituent minerals and their grain boundary and consider the correlation of the fracture between the constituent minerals and the rock composed of them. Then, we aim to clarify the mechanism of fracture progress of macroscopic rocks, which are composites, based on a microscopic viewpoint. Furthermore, we will find the correlation between the hardness and fracture toughness values of each mineral, leading to the construction of a new index that clearly indicates the relative strength of minerals.

2 PROPERTIES OF INADA GRANITE

In this study, Inada granite (Ibaraki, Japan) is used for specimen. The major mineral composition of Inada granite consists of quartz (36%), plagioclase (32%), K-feldspar (28%), and biotite (4%) (Matsuki et al., 1999). The average grain size of quartz is about 3-4 mm, that of plagioclase and K-feldspar is about 2-3 mm, and that of biotite is generally less than 1 mm.

In addition, from the mechanical tests conducted for this study, the uniaxial compressive strength is 67.3 MPa, the tensile strength is 6.2 MPa, the Young's modulus is 39.5 GPa, and the Poisson's ratio is 0.19.

3 MACROSCOPIC FRACTURE TOUGHNESS TEST

The Semi-Circular Bend (SCB) test was conducted to determine the macroscopic fracture toughness of Inada granite. The SCB test is one of the ISRM-suggested method for determining the mode I static fracture toughness of rock (Kuruppu et al., 2014). In this study, we prepared specimens of five sizes (15 mm $\leq R \leq$ 80 mm) shown in Figure 1 to investigate the size effect of macroscopic fracture toughness of Inada granite as well. Experiments were performed with 3-4 specimens of each size.

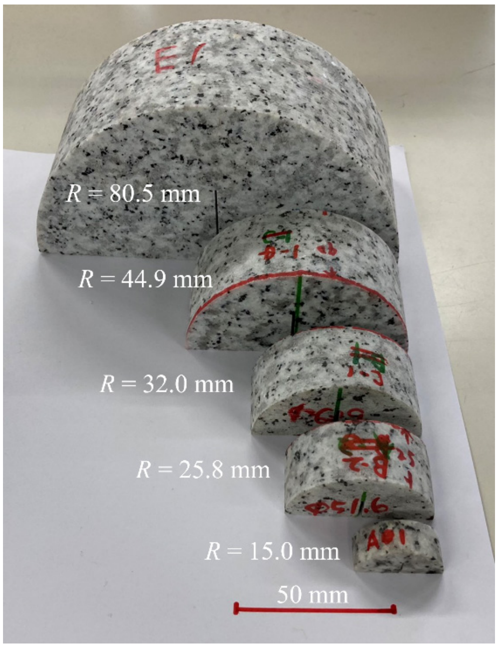

Figure 1. Specimen for SCB test.

Figure 2 shows the results of the macroscopic fracture toughness determined by the SCB tests. The results of R = 44.9 mm size specimen are not shown because they were unsatisfactory. Although there are variations in the fracture toughness for each size, the overall trend indicates that there is a size effect. The solid black line in this figure is a curve based on Bažant size effect law (Bažant et al., 1991). The results of this experiment are well fitted to this Bažant curve. Therefore, the convergence value of the fracture toughness of Inada granite used in this experiment was estimated to be 1.20 MN/m$^{3/2}$. Hereafter, this value is called the representative fracture toughness, $K_{IC\_Inada\text{-}G}$.

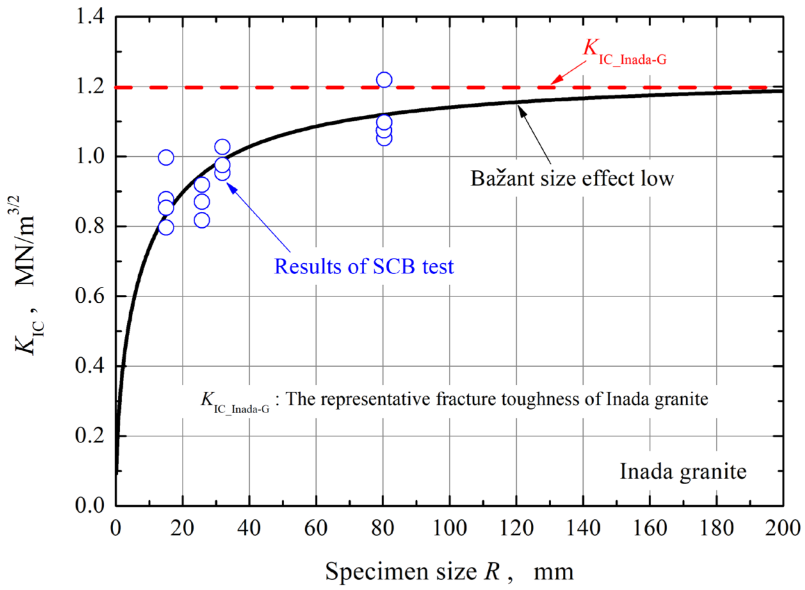

Figure 2. Relation between macroscopic fracture toughness and specimen size for Inada granite.

4 MICROSCOPIC FRACTURE TOUGHNESS TEST

The Microscopic fracture toughness (MFT) test was performed by the method proposed by Kataoka et al. (2018).

Figure 3 shows the MFT test equipment. The MFT test equipment consists of an observation unit consisting of a microscope and a monitor, and a measurement unit consisting of a loading pin, a load cell, an actuator, and so on. The microscope can be adjusted from 250 to 2500 times. The loading status of the micrometer size specimen by the loading pin can be observed in real time on a monitor connected to the microscope.

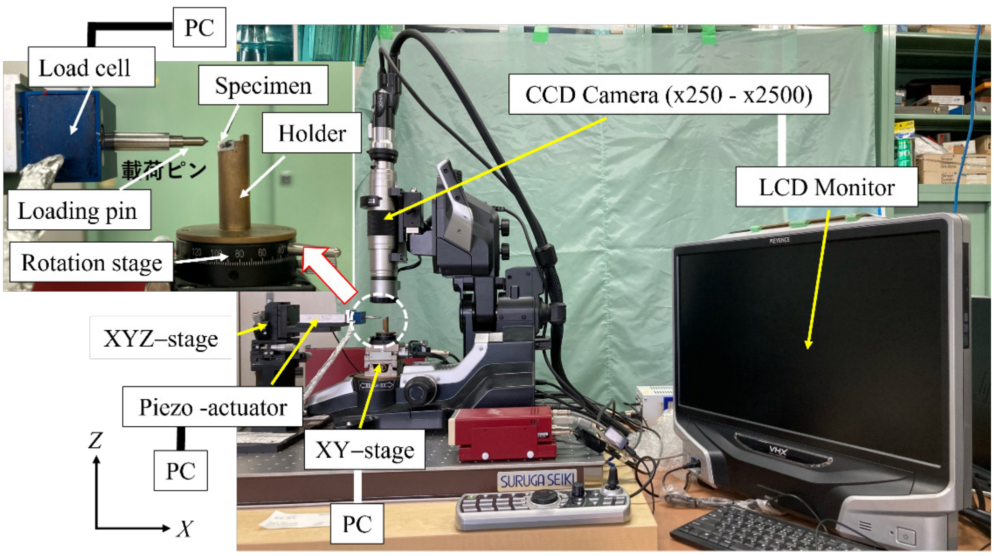

Figure 3. View of the MFT test equipment.

The micrometer size specimen is a cantilever type with an artificial notch as shown in Figure 4. The corners of a rectangular parallelepiped sample of 3 mm × 5 mm × 7 mm of Inada granite were processed by FIB (Focused Ion Beam) machining according to the procedure shown in the Figure 5. Prior to FIB machining, the corners of the rectangular parallelepiped sample were mapped by EPMA

(Electron Probe Micro Analyzer) to identify the position of each mineral. To evaluate the scale dependence of the mineral MFT, we conducted MFT tests using three cantilevered specimens (Type A; B = 10 μm × W = 10 μm × L = 40 μm, Type B; 20 μm × 20 μm × 40 μm, Type C; 20 μm × 20 μm × 80 μm). The size of the specimen for the mineral grain boundary MFT test was $B \approx 10$ μm × $W \approx 10$ μm × $S \approx 30$ μm because machining is difficult.

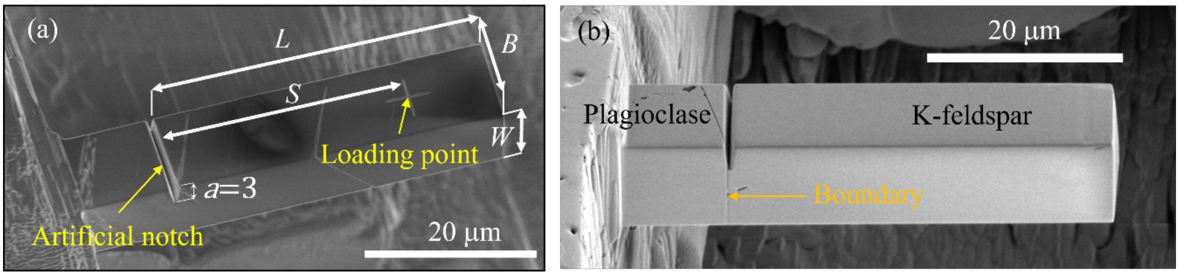

Figure 4. SEM image of the micro-sized specimen. (a) Biotite, (b) Specimen with boundary.

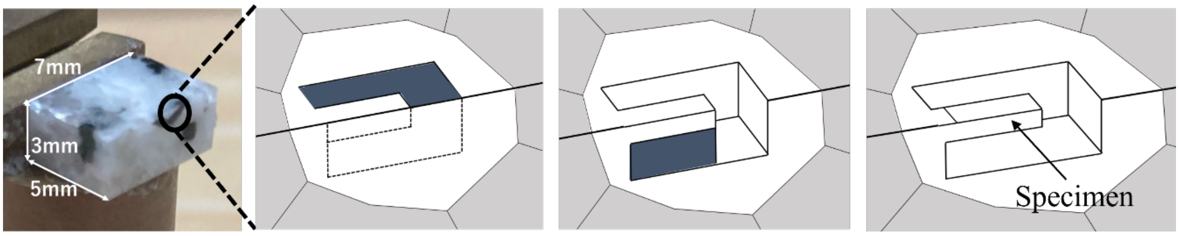

Figure 5. Procedure of preparation of micro-sized specimen using FIB.

The mode I fracture toughness, K_{IC}, was obtained from the maximum load P_{max} using the following equation (Murakami, Y., 1987).

$$K_{\mathrm{IC}} = \frac{6P_{max}S\sqrt{\pi a}}{W^2B}F\left(\frac{a}{W}\right) \tag{1}$$

where:
a = depth of the artificial notch,
W = thickness of the cantilever beam,
B = width of the cantilever beam,
S = distance between the loading point and the notch, and
Y = function of dimensionless notch length a/W, as follows:

$$Y\left(\frac{a}{W}\right) = 1.122 - 1.40\left(\frac{a}{W}\right) + 7.33\left(\frac{a}{W}\right)^2 - 13.08\left(\frac{a}{W}\right)^3 + 14.0\left(\frac{a}{W}\right)^4 \tag{2}$$

Figure 6 shows the results of the fracture toughness, K_{IC}, of mineral and mineral grain boundary from MFT tests. The fracture toughness of each mineral does not show the size effect. The mean fracture toughness of each mineral is 0.86 for quartz, 0.64 for plagioclase, 0.60 for K-feldspar, and 0.11 for biotite. There is not much difference between K-feldspar and plagioclase. The representative value of MFT of all minerals composing the Inada granite, $K_{IC\_mineral}$, is calculated to be 0.69 from the mean fracture toughness and composition ratio of each mineral. The fracture toughness of the mineral grain boundary is smaller than that of minerals. Although it is doubtful whether the conditions of the grain boundaries used in the experiment were the same, the results of this study may help introduce the parameters for intragranular fracture and intergranular fracture into numerical simulation. $K_{IC\_mineral}$ is about half smaller than $K_{IC\_Inada\text{-}G}$. This means that the macroscopic fracture toughness of rock cannot be directly related to the fracture toughness of constituent minerals. However, macroscopic fracture toughness of rock may be affected by the mineral with the highest fracture toughness among the constituent minerals of the rock (grain size, arrangement, etc.).

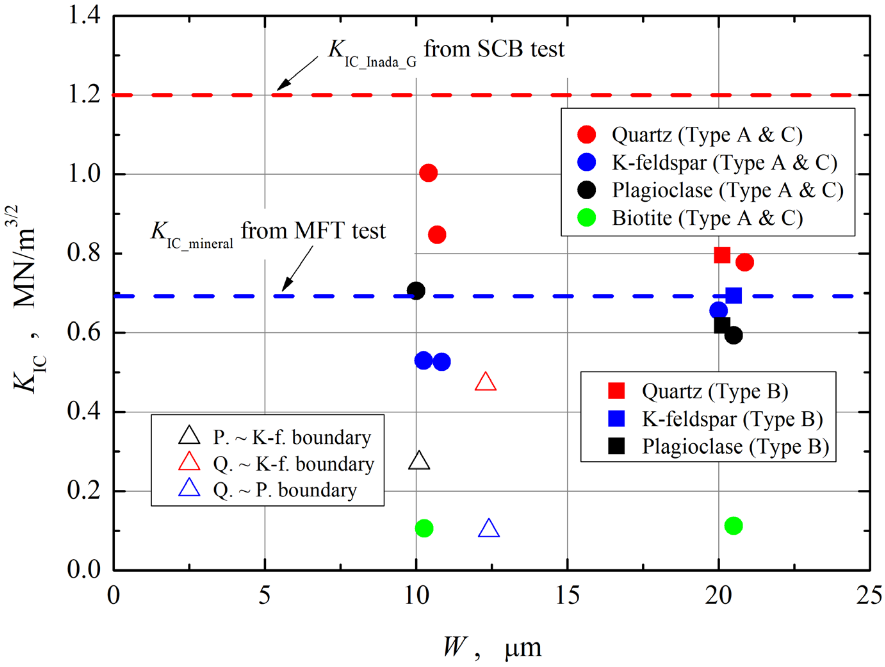

Figure 6. The results of the fracture toughness, K_{IC}, of mineral and mineral grain boundary from MFT tests.

5 CORRELATION BETWEEN MFT AND HARDNESS OF MINERALS

Figure 7 shows the relation between the MFT of minerals and Vickers hardness. The broken line in this figure is the result of linear approximation. Since the correlation coefficient is high, it can be seen that there is a strong correlation between the two. Using this relationship, it may be possible to estimate fracture toughness from Vickers hardness (or vice versa).

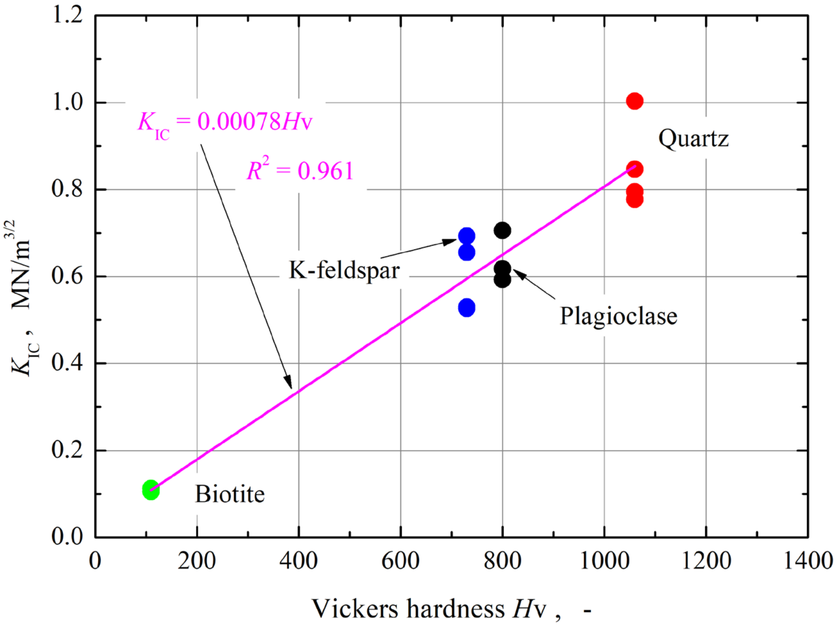

Figure 7. Relation between the MFT of minerals and Vickers hardness (Adebayo, B & Okewale, I.A. 2007).

6 CONCLUSIONS

The main results of this study are as follows.

1. The MFT of mineral was in the order of quartz, plagioclase, K-feldspar, and biotite from the largest value. We found no significant scale dependence for the MFT itself.
2. The representative value of MFT of all minerals composing the Inada granite, $K_{IC\_mineral}$, is about half smaller than the representative fracture toughness of Inada granite, $K_{IC\_Inada\text{-}G}$.
3. The mineral grain boundary MFT was smaller than the mineral MFT.
4. The results of this study may help introduce the parameters for intragranular fracture and intergranular fracture into numerical simulation.
5. It was suggested that macroscopic fracture toughness of Inada granite may be controlled by quartz, which has a large composition ratio and large MFT.
6. It was clarified that the MFT of each mineral has a positive correlation with the Vickers hardness.

ACKNOWLEDGEMENTS

This study was partially supported by the Japan Society for the Promotion of Science (JSPS) through Challenging Research (Pioneering) (No. 21K18200). The authors would like to thank Hiroki Asai (Master student at the time), Masanori Tashiro (for EPMA) and Yumiko Kodama (for FIB). The MFT equipment was lent free of charge from Kumamoto University.

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