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Influence of firing temperature on frost resistance of roofing tiles

Kiyohiko Ikeda [a,*], Hyung-Sun Kim [b], Koichi Kaizu [a], Atsushi Higashi [c]

[a] Department of Mechanical Systems Engineering, Miyazaki University, 1-1 Gakuenn Kibanadai Nishi, Miyazaki 889-2192, Japan
[b] School of Applied Materials Engineering, Sunchon National University, 315 Maegok-dong, Sunchon 540-742, South Korea
[c] Graduate School of Miyazaki University, Miyazaki, Japan

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Abstract

In cold districts, prevention against frost damage of roofing tiles caused by the expansion due to the freezing of water absorbed in the numerous pores in the tile material has becomes a serious problem. It is known that the frost damage is closely connected to the porosity and water absorbing capacity of the roofing tile. Both the porosity and water absorbing capacity of the material can be controlled by the firing temperature. In this work, in order to examine the effect of firing temperature on the frost resistance, tiles fired at different temperatures of 900–1200 °C were fractured by four point bending after applying freezing treatment at low temperatures of −10, −30 and −50 °C. At any firing temperature up to 1100 °C, both fracture stress and fracture toughness increased with the firing temperature, but they dropped above 1100 °C. Furthermore, an obvious decrease in fracture stress with decrease in freezing temperature was observed for the samples fired at 1200 °C. Thus, it was concluded that the roofing tile fired at temperatures of up to 1200 °C forming mullite with a glass phase from the reaction with quartz and feldspar could minimize the frost damage.

Keywords: Roofing tile; Frost damage; Porosity; Freezing; Damage; Firing; Tiles

1. Introduction

Roofing tiles have been widely used on the roofs of detached houses. But, when they are used in cold districts, frost damage which is caused by the freezing of water which has permeated into the numerous pores existing in that material become a serious problem.[1–4] To describe in details, when water contained in some of these pores following rainfall freezes into ice due to a decrease of temperature, local high tensile stress will arise around these pores, and micro-cracks will be generated. These micro-cracks will grow and join with each other until the final fracture occurs. Thus, it is necessary to clarify the effect of the porosity on the brittle fracture as a measure to counter frost damage.

The frost resistance is influenced by the pore-size, pore type, and pore distribution, which depend on firing temperature.[2–6] The roofing tile is a complex, heterogeneous material consisting of quartz, feldspar, and numerous pores. It is considered that the change of the constituent minerals and microstructure of the bodies with a densification process influences the frost resistance.[7–13]

In this work, roofing tiles fired in the range of 900–1200 °C were used as the samples to examine the effect of firing temperature on the frost resistance. Regarding the frost resistance of tiles, we did not apply cycling tests such as DIN 52253 for roofing tiles and EN 202 for floor tiles, which are known to be conventional methods. These samples were fractured by four point bending after applying freezing treatment at low temperatures of −10, −30 and −50 °C. The effects of firing temperature and freezing temperature on fracture stress and fracture toughness were examined.

2. Experimental procedure

2.1. Materials and specimens

The materials are unfired roofing tiles formed into a roof shape, which consist of kaolin, quartz and feldspar. The chemical content is shown in Table 1. The test samples cut from the roofing title were fired at 900, 1000, 1100 and

* Corresponding author. Tel.: +81-985-58-7292; fax: +81-985-58-2876.
E-mail address: t0d106u@cc.miyazaki-u.ac.jp (K. Ikeda).

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Table 1
Chemical compositions of roofing tile (wt.%)

SiO_2	Al_2O_3	Fe_2O_3	K_2O	MgO	Na_2O	TiO_2
56.20	20.30	3.99	2.98	1.37	1.01	0.75
CaO	BaO	SO_3	MnO	P_2O_5	ZrO_2	Cl
0.71	0.16	0.12	0.09	0.08	0.02	0.02
Cr_2O_3	SrO	Rb_2O	ZnO	Y_2O_3	NiO	Ig loss
0.02	0.02	0.01	0.01	tr	tr	5.20

1200 °C. After that, each sample was polished to a size of 3 mm × 4 mm × 40 mm. The tile porosity and pore-size were measured by the mercury method (Pore-sizer 9320, Micromeritics Inc.). The SEM observations (S-4100M, Hitachi Ltd.) were conducted to examine the microstructure of tiles fractured by bending.

2.2. Experimental method

All specimens were immersed in dry ice with ethanol for freezing at low temperatures of −10, −30 and −50 °C after being immersed in a distilled water at room temperature for 24 h. The specimens, frozen by cooling to the respective low temperatures, were returned to room temperature. Each of the 10 specimens was fractured by four point bending with an inner span of 10 mm and outer span of 30 mm under a 0.05 mm/min cross head speed. Fracture toughness was also measured by the Single Edge V-Notched Beam (SEVNB) method,[14] where V-notches at the center of the specimen's tensile surface were cut before testing. The V-notches could be produced at the bottom of a straight notch cut to a depth of approximately 0.2 mm by using a razor blade sprinkled with diamond paste. The bending tests were conducted with a fast cross head speed of 10 mm/min.

The fracture toughness K_{IC} can be computed using the following formula:[14]

$$K_{IC} = \frac{F}{B\sqrt{W}} \frac{S_1 - S_2}{W} \frac{3\sqrt{\alpha}}{2(1-\alpha)^{1.5}} Y, \qquad (1)$$

where F, S_x, and a are fracture load, span, and notch depth, respectively; B and W are specimen width and specimen height, respectively; $\alpha = a/W$; Y is calculated using the following equation:[14]

$$Y = 1.9887 - 1.326\alpha - (3.49 - 0.68\alpha + 1.35\alpha^2) \times \alpha(1-\alpha)(1+\alpha)^{-2}. \qquad (2)$$

3. Results and discussions

3.1. Dependences of fracture stress and fracture toughness on firing temperature

Figs. 1 and 2 show the variation of the fracture stress and the fracture toughness as a function of firing temperature for

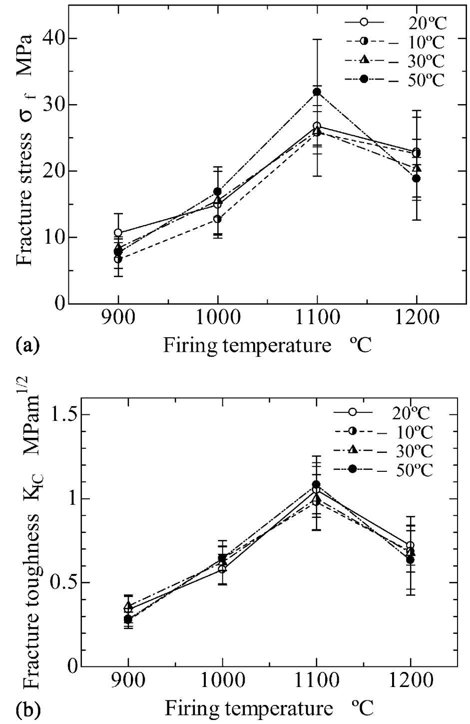

Fig. 1. Dependences of (a) fracture stress and (b) fracture toughness on firing temperature.

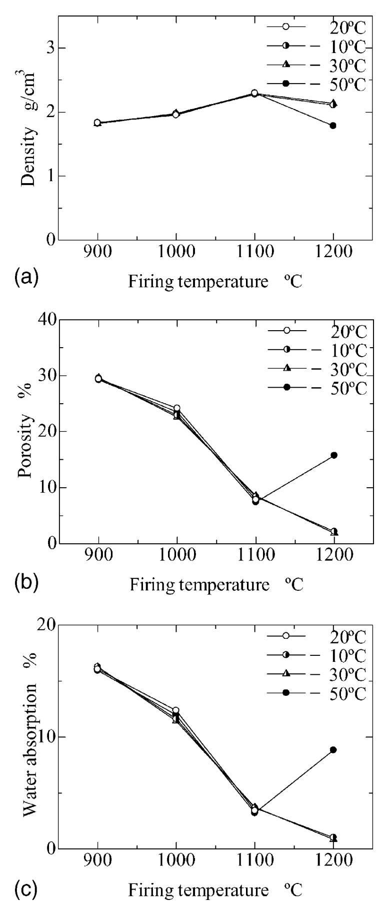

Fig. 2. Change of (a) density, (b) porosity and (c) water absorption with firing temperature.

the roofing tile specimens subjected to freezing treatments, in which the data are also plotted for the specimens subjected to pretreatment by submerging into water at 20 °C. Error bars are standard deviations. For any freezing temperature, the fracture stress increased monotonically with firing temperature up to 1100 °C, but decreased above 1100 °C. The tendency was more significant in the case of the specimen frozen at the lowest temperature of −50 °C. The fracture toughness also showed almost the same tendencies as the fracture stress, but there was no significant difference at different freezing temperatures. The variation of the fracture stress and the fracture toughness by firing temperature is related to the firing conditions used to control the microstructure of tiles to produce dense materials by the reaction of feldspar and quartz.[7,15]

3.2. *Effect of firing temperature on microstructure of roofing tile*

Fig. 2(a)–(c) shows the effect of firing temperature on density, porosity and water absorption of tiles for each freezing temperature. For all freezing temperatures, the density increased with temperature up to 1100 °C, but it dropped above 1100 °C. On the other hand, both porosity and water absorption decreased monotonically up to 1100 °C, but these rose for the freezing temperatures of −10 and −30 °C and dropped for room temperature and the freezing temperature of −50 above 1100 °C. It is reported that there is a correlation between pore distribution and frost resistance.[2–6,16] Fig. 3(a)–(d) shows the data of pore-size distribution, which was measured by the mercury intrusion method. The results are plotted with the diameter of pore as abscissa against pore volume per unit weight, which was calculated from the data of accumulated pore-size distribution, as ordinate. Pore-size distribution is not significantly affected by freezing temperature but is affected by firing temperature. For samples fired at a temperature of 900 °C, the distribution has a peak in pore-size of 0.1–0.5 μm diameter and pore-size of 100 μm diameter. At 1000 °C, the peak of the pore-size of 0.1–0.5 μm shifts to 1–2 μm and the height of the peak increases, but the height of the peak at 100 μm decreases. At 1100 °C, the height of the peak at 1–2 μm abruptly decreases and the accumulated pore distribution also decreases. However, at 1200 °C, the peak for the pore-size of 1–2 μm shifts again to a pore-size of 0.1–0.5 μm and the accumulated pore distribution increases. It has been reported by Ravaglioli[16] that the roofing tiles with pore-size of 0.25–1.4 μm suffer from more frost damage. Thus, taking the foregoing report into consideration, it is expected that the roofing tile fired at 1200 °C would exhibit frost damage.

Fig. 4(a)–(d) shows XRD results for the raw material before firing and for the material after firing at each temperature. It was found that the proportion of each component is dependent on the firing temperature and composition of the raw material. The components of the raw material are kaolin, quartz and albite feldspar. The feldspar amount decreases with firing temperature and mullite with glass phase is formed from the reaction with quartz and feldspar at 1100 and 1200 °C, respectively.[7,15] Furthermore, the portion of the glass phase increases with increasing temperature.

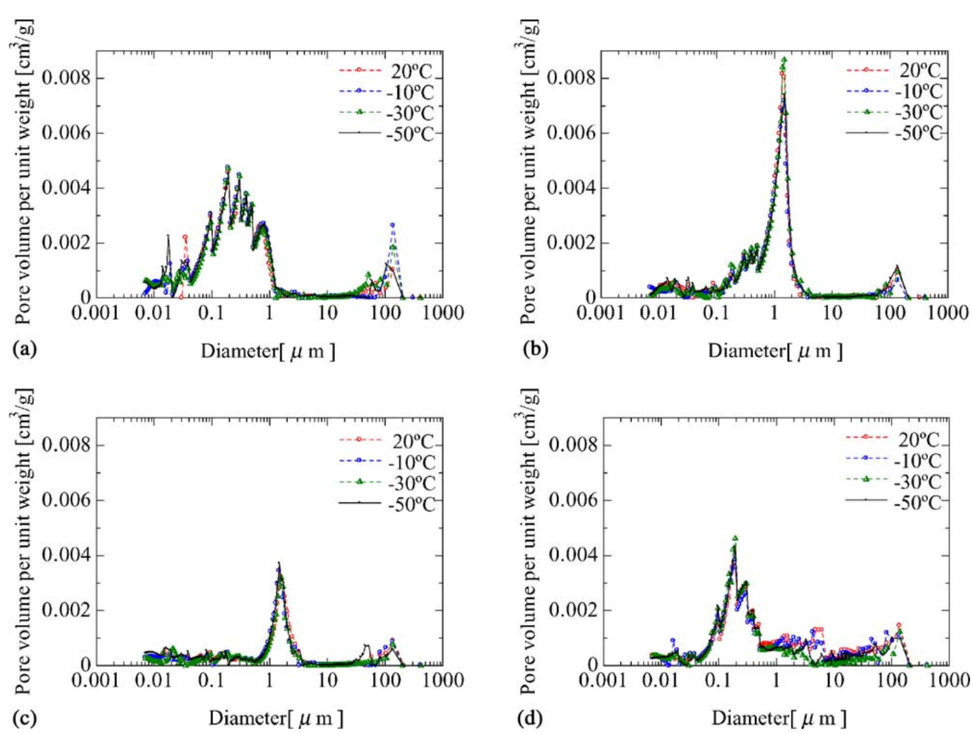

Fig. 3. Pore-size distribution of roofing tiles fired at respective temperatures of (a) 900, (b) 1000, (c) 1100, and (d) 1200 °C.

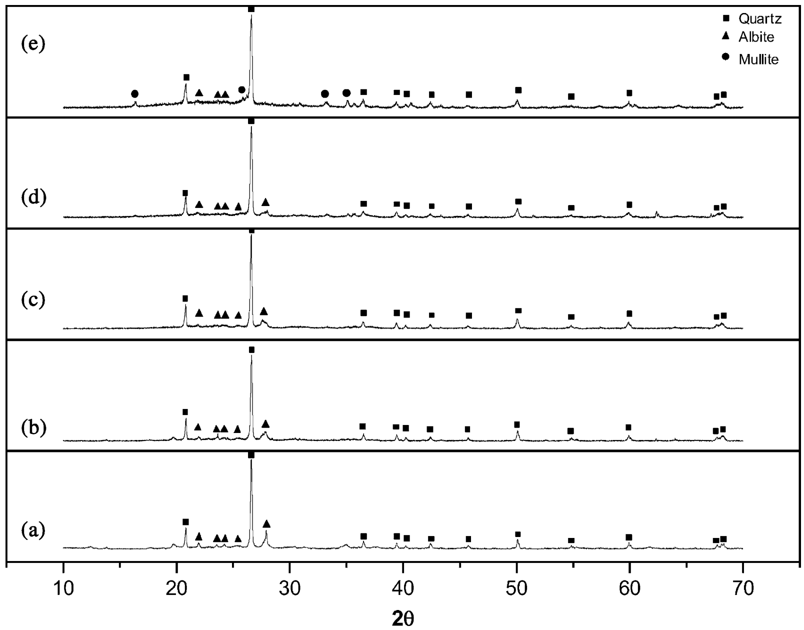

Fig. 4. X-ray diffraction profiles for roofing tile: (a) raw materials before firing, (b) after firing at 900 °C, (c) after firing at 1000 °C, (d) after firing at 1200 °C; (■: quartz, ▲: albite feldspar, ●: mullite).

From these facts, the following can be deduced. There are numerous many open macro-pores in tiles fired at low temperatures and these pores become closed micro-pores because of the increase of the glassy phase formed from the reaction with quartz and feldspar with the increase of firing temperature. This causes the increase in density and the decrease in porosity with increasing temperature. On the other hand, the drop in density and increase in porosity at 1200 °C seem to be due to the expansion of air frapped in the micro-pores. In freezing at −10 or −30 °C, many cracks generated by frost damage enable the air trapped in pores to escape, resulting in the drop in porosity. On the contrary, for the lowest freezing temperature of −50 °C, the rise in porosity at 1200 °C was observed in the same way as for room temperature. This seems be due to the fact that more micro-cracks occurred by frost damage at the lowest temperature of −50 °C. Fig. 4(a)–(d) shows the SEM observations of the sample for each firing temperature. Clearly, more closed micro-pores were observed in the samples fired at 1100 and 1200 °C than at the other low firing temperatures, and the pore-size at 1200 °C was larger than that at 1100 °C.

3.3. Dependences of fracture stress and fracture toughness on freezing temperature

Figs. 5 and 6 show the variation of the fracture stress and fracture toughness, respectively, as a function of freezing temperature fired at 900, 1000, 1100 and 1200 °C. In all of the samples except for those at temperatures below 1100 °C, the fracture stress somewhat decreased with decreasing freezing temperature. This tendency was clearly evident for the sample fired at 1200 °C. As stated above, a number of macro open and blind-alley pores are produced in tiles fired at temperatures of below 1000 °C, and more closed micro-pores are produced in the tile fired at high temperatures above 1200 °C, in which feldspar produces glass and thus pore reduces the pore-size. It is said that frost damage in tiles is due to the expansion of limited pore volume due to the conversion of water contained in them into ice.[3] The slight frost damage at firing temperatures below 1100 °C, that is, the small decrease in fracture stress with a decrease in freezing temperature, seems to be due to a number of macro open and blind-alley pores produced at low temperatures and the clear frost damage at 1200 °C, that is, the clear decrease in fracture stress with decreasing freezing temperature seems to be due to the many closed pores produced at high temperatures. On the other hand, no distinct decrease of the fracture toughness with lowering of freezing temperature could be recognized in any of the samples. The pre-compressive stress produced around large grains such as those of quartz by the expansion of water due to freezing could increase the fracture resistance. This fact indicates that bonding between larger quartz and the matrix surrounding the quartz increased with the decrease in freezing temperature. The above slight frost damage in fracture toughness is due to the fact that the degradation by the occurrences of micro-cracks and the effect of increase in fracture resistance by the strong bonding between quartz and the matrix compensate for each other.

3.4. Relationship between fracture stress and porosity

It is important to consider porosity as well as pore-size distribution of tiles in order to understand the fracture

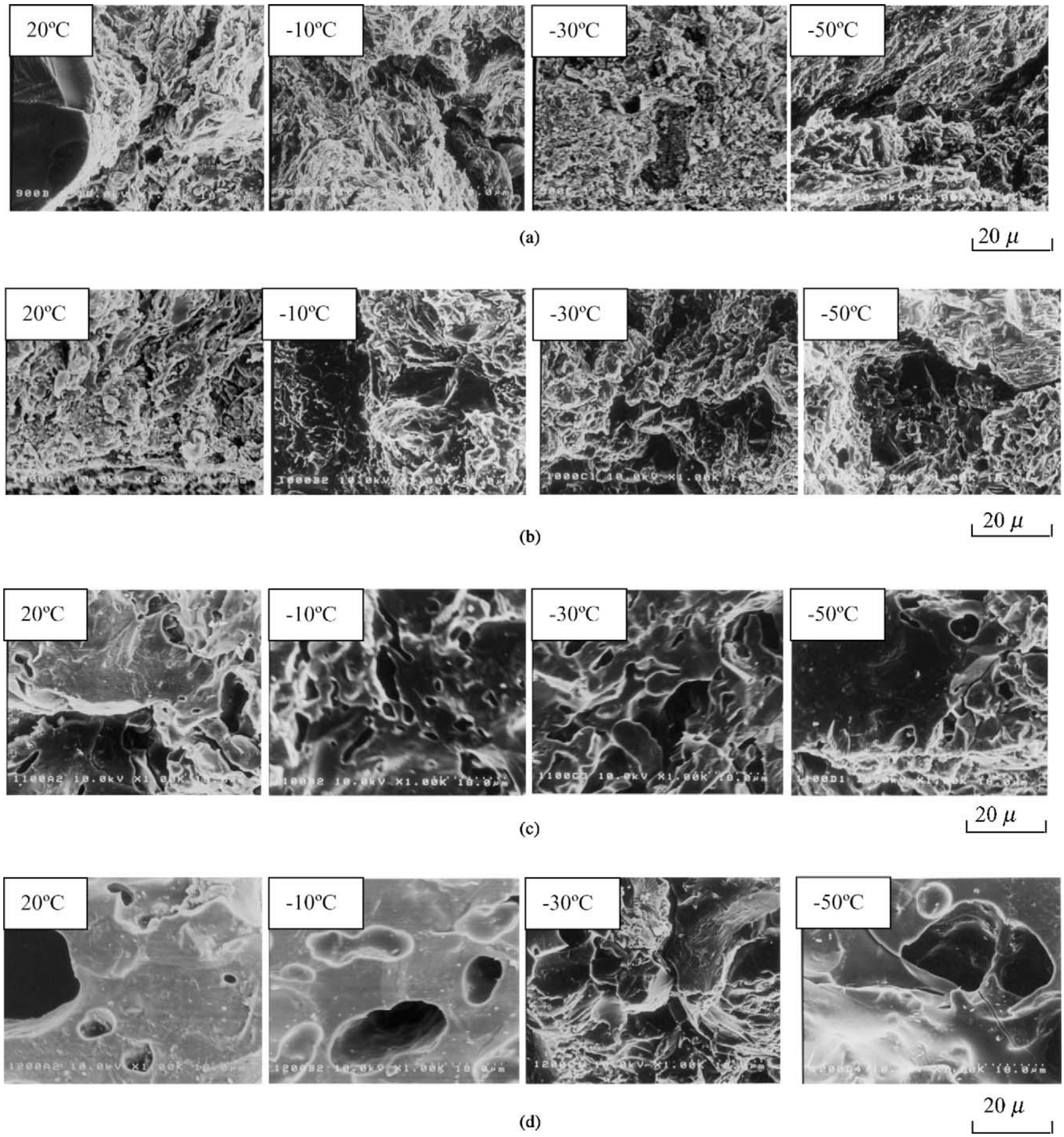

Fig. 5. SEM observation of samples for respective temperatures of (a) 900, (b) 1000, (c) 1100, and (d) 1200 °C.

phenomena at low temperatures. Fig. 7 shows the effect of porosity on fracture stress for each freezing temperature. The influence of volume fraction of porosity (P) on the strength can be expressed empirically as follows:

$$S = S_0 \exp(-bP), \qquad (3)$$

where S_0 is strength at $P = 0$, P is volume fraction porosity, and b is an empirical constant.[17–19] S_0 and b obtained by regression analysis are shown in Table 2. The strength of the tiles assuming free pores, S_0, decreased from 42.2 to 27.2 MPa with decreasing temperature from room temperature to −30 °C, but at −50 °C, the strength S_0 recovered to 50.4 MPa. The decrease in the strength in the temperature range from 20 to −30 °C seems to be due to the freezing effect, that is, the occurrences of micro-cracks by expansion of pore volume because of the transformation to ice of water included in the pores. Below −30 °C, the strength will be improved. An increasing

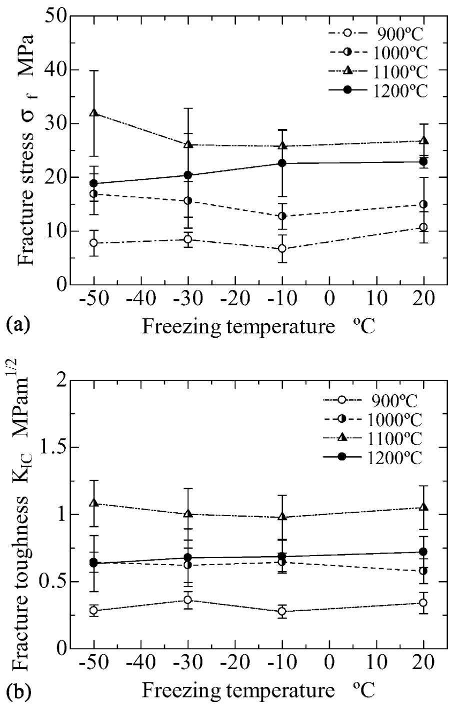

Fig. 6. Dependences of (a) fracture stress and (b) fracture toughness on freezing temperature.

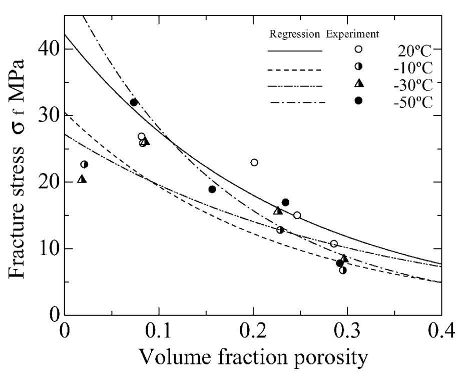

Fig. 7. Effect of porosity on fracture stress for freezing temperature.

strength at a low temperature of −50 °C seems to be due to the fact that the crack propagation resistance increases by compressive stress occurring around a number of quartz with a larger size because of an expansion of pore volume due to the transformation to ice of water confined in the pores.[20]

Table 2
Empirical relation on porosity for freezing temperature

Temperature (°C)	S_0 (MPa)	b
20	42.2	4.25
−10	30.5	4.55
−30	27.2	3.29
−50	50.4	5.82

4. Conclusions

For the samples fired below 1100 °C, a slight decrease in fracture stress with lowering in freezing temperature, but for the samples fired at 1200 °C, the obvious decrease in fracture stress was observed. It seems to be due to a number of macro open and blind-alley pores, and more closed micro-pores produced, respectively, at low and high temperatures. There is no distinct decrease of the fracture toughness with lowering of freezing temperature in all of the samples. The slight frost damage in fracture toughness is owing to the degrading effect by the occurrences of micro-crack and the effect increasing the fracture resistance by the strong bonding between quartz and the matrix will compensate each other. The roofing tile fired at temperature up to 1200 °C forming mullite with glass phase from the reaction with quartz and feldspar could minimize the frost damage.

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