

SHINAGAWA TECHNICAL REPORT

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品川技報

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要 旨

珪石れんがの常温と高温における疲労破壊について調査した。その結果、珪石れんがの1470Kにおける疲労寿命は常温での疲労寿命に比較して大幅に優れることがわかった。高温における疲労寿命の延長メカニズムについて詳細に検討した結果、高温ではれんが内部に形成される微量な液相の存在が疲労破壊寿命の増加と関連性があると推定された。さらに液相と固層との濡れ性が高温での疲労破壊寿命に依存しており、両者の濡れ性が悪い場合には高温で疲労には優れると推定された。

Abstract

The fatigue failure behavior of silica brick refractory materials was investigated at room temperature and high temperature. It was found that the fatigue failure life of the silica bricks increased significantly at temperatures over 1473K compared with that in the fatigue failure test at room temperature. The reason for this improved fatigue fracture life at high temperatures was examined in detail. It was deduced that longer fatigue fracture life of silica bricks at high temperatures is related to the generation of a slight amount of liquid phase. The length of the fatigue fracture life of the bricks was estimated to depend on the wettability between the solid phase and liquid phase, with poor wettability resulting in extended fatigue fracture life.

1. はじめに

珪石れんがは主にコークス炉などに用いられる耐火物で、この耐火物には長期にわたる耐用性が要求される。コークス炉向け珪石れんがに求められる特性のひとつに耐熱スポール性がある。一般に製鉄、製鋼の耐火物設備は、溶鉄、溶鋼を含む原料の装入、排出時に大量の熱の放出を伴う。このような熱の出入りを繰り返し行くとウェア耐火物、永久耐火物内部でその熱の出入りに応じて熱応力が発生し、その結果、耐火物内部に亀裂が発

1. Introduction

Silica brick is a refractory material which has been used mainly in coke ovens, and long-term durability is necessary in this refractory. One property which is required in silica bricks for coke ovens is high thermal shock resistance (spalling resistance). In general, a huge amount of heat is radiated from ironmaking and steel-making equipment during charging or discharging of raw materials, including molten metal.

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生，進展して破壊が起こることが予想される。このような破壊は瞬間的な温度変化によって生じる熱衝撃破壊とは異なり，長期的なスパンで起こると考えられる。それゆえ，耐火物内に発生して破壊に至るまでの期間を評価するためには，繰り返し熱負荷と耐火物の長期的なスパンでの破壊との関連性を調査する必要がある。さらに，耐火物寿命の向上には，高温条件での亀裂進展機構の詳細な解明が要求される。一般的に製鉄，製鋼の耐火物設備に用いられる耐火物として，亀裂が発生，進展しにくい材質を用いることにより高寿命が得られる。耐火物の特性を改善するには2つのアプローチ，すなわち熱応力発生を低減させるための特性改善，および亀裂発生，進展に影響を与える特性の改善，が考えられる。これまでの研究のほとんどは前者，熱応力低減に注力しており，材料の弾性率や熱膨張係数を制御することによって耐火物の改善がなされてきた^{2),3)}。しかしながら，これらの研究は熱膨張率などの物性値の変化が，破壊強度など材料の機械的特性に影響を及ぼす可能性を調査しておらず，耐火物の破壊強度に及ぼす繰り返しの熱の出入りによって生じる応力の影響に関する適切な検討が十分にできていない。その結果，周期的な熱の変化により，どのくらいの頻度で破壊に至るかまでの検討に関する情報は不十分であった。それゆえ，繰り返し応力負荷での材料の破壊を評価する手段として，疲労破壊試験に着目した。

このような周期的な応力付与の条件下での破壊挙動の観点から疲労破壊をみると，セラミックスやコンクリートの分野では多くの研究例がある^{4)-11), 16)}。しかし，耐火物技術ではほとんど見られない¹²⁾⁻¹⁵⁾。それゆえ，著者はその第一段階として，繰り返し応力を材料に直接印加し，その条件下での耐火物の疲労寿命を評価することで，材料の熱膨張率や弾性率と分離して，耐火物材料内の亀裂進展を調査した。過去に著者は，常温および高温で繰り返し応力印加条件のもとMgO-Cれんがの疲労破壊挙動を調査した。そして，疲労試験の結果と破壊力学的考察に基づき，疲労破壊挙動と熱スポール破壊との関連性を検討した¹⁵⁾。その結果，

As these charging and discharging processes are repeated, the wear refractories and safety brick undergo repeated and varying thermal stresses, which cause progressive cracking in the refractory materials, in some cases resulting in fracture. Refractories must possess adequate mechanical properties to tolerate this periodical thermal stress. These types of damage are not short-term phenomena which occur instantaneously, but rather, are the phenomena which develop progressively over an extended period of time. Thus, in order to estimate the period from crack initiation to fracture of refractories, it is necessary to investigate the relationship between cyclical inputs and discharges of heat and fracture of refractories over a long-term span. Moreover, in order to improve refractory life, a detailed clarification of the mechanism of crack growth under high temperature conditions is also required.

Generally, longer life can be obtained if materials which possess resistance to crack initiation and growth are used as refractories for ironmaking and steelmaking equipment. Two approaches to improving refractory properties are conceivable, that is, improvement of properties in order to reduce thermal stress, and improvement of properties which affect crack initiation and growth. The focus of most past studies was on reduction of thermal stress. Although improvement of refractories has been achieved by controlling the elastic modulus and coefficient of thermal expansion of the material,^{2),3)} these studies did not examine the possibility that changes in properties such as the thermal expansion ratio might affect mechanical properties such as the modulus of rupture. Consequently, adequate consideration was not given to the effect of the thermal stresses generated by cyclical heating and cooling on the modulus of rupture of refractory materials. As

動的疲労破壊に強い耐火物が耐熱スポール性に優れる理由のひとつになっていることを明らかにした。このように、耐火物材料の疲労破壊を検討することは、材料の耐熱スポール性を評価するためにも重要である。そこで今回は、 SiO_2 を主に含有し、カーボンを含まない焼成れんがに着目し、珪石れんがの常温、高温での繰り返し圧縮応力下での疲労破壊挙動を調査した。

2. 実験方法

2. 1 疲労試験

実験に用いた珪石れんがの組成、鉱物相、常温、高温での圧縮強度の一覧をTable 1に示す。試験片のサイズは20mm立方体とした。

载荷方法をFig.1に示す。荷重印加方法は、緻密質のファインセラミックス製のシリンダを通じ

Table 1 Chemical composition, phase composition and mechanical strength of materials used in this study

		Silica brick
Chemical composition	(% Al_2O_3)	0.9
	(% SiO_2)	95.4
	(% CaO)	0.3
	(% MgO)	2.4
Phase (from XRD)		SiO_2 (cristobalite) SiO_2 (tridymite) SiO_2 (quartz)
Apparent porosity (%)		18.7
Bulk density (g/cm^3)		1.88
Sample size (mm)		20×20×20
Compressive stress σ_c (MPa)	298K	45.8
	1273K	41.1
	1473K	18.2
	1673K	9.3

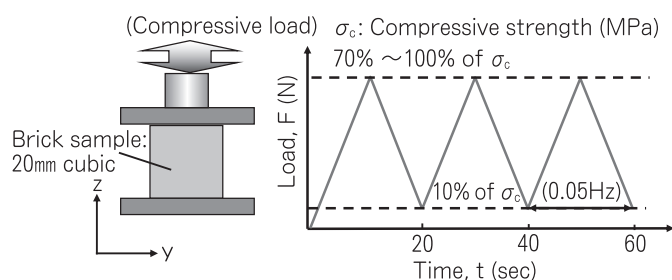


Fig.1 Experimental apparatus for fatigue fracture test and loading pattern.

a result, information on how many repetitions of these thermal changes a refractory material can withstand before fracture was inadequate. Therefore, attention was focused on fatigue failure as a means of evaluating the fracture of materials under cyclical stress loading.

Many studies of ceramics and concrete^{4)-11), 16)} have investigated fatigue failure from the viewpoint of fracture behavior under cyclical stress loading, but there have been few studies on refractory technology¹²⁾⁻¹⁵⁾. Therefore, as a first step, the author investigated the crack growth behavior in refractory materials separately from the thermal expansion ratio and elastic modulus by conducting fatigue failure tests in which cyclical stress was loaded directly on the material, and evaluated the fatigue life of refractories under cyclical stress. In previous research, the author investigated the fatigue failure behavior of MgO-C brick under cyclical stress at room temperature and high temperature¹⁵⁾. The relationship between fatigue failure behavior and thermal spalling fracture has been examined based on the results of the fatigue failure test and discussion in terms of fracture mechanics. It was found that a brick which had strength against dynamic fatigue failure also possessed excellent resistance against thermal spalling¹⁵⁾. Thus, it is important to investigate the fatigue failure of refractory materials in order to evaluate their thermal spalling resistance. The present study investigates the fatigue failure behavior of silica brick under cyclical compressive stress in room temperature and high temperature atmospheres, focusing on fired bricks containing mainly SiO_2 but not containing carbon.

2. Experimental Procedure

2. 1 Fatigue failure test

Table 1 shows the chemical composition,

て、圧縮荷重を三角波の波形で繰り返し付与させた。載荷荷重値については、疲労試験を実施するに先立ち、静的圧縮試験を行ない、各種れんが、各種条件での圧縮破壊強度を測定した。各温度(298K, 1473K, 1673K)の圧縮破壊強度もTable 1に示す。圧縮破壊強度の導出方法は以下の通りである。試験片に一定載荷速度(0.5mm/min)で圧縮加重を印加し、試験片の断面積で破壊したときの荷重値を除した値を用いた。疲労試験は繰り返し圧縮荷重を大気雰囲気中で印加しつつ実験を行った。実験は平均圧縮破壊強度値を100%としたとき、70~100%に相当する応力値を、下限値については圧縮破壊強度の10%と固定させて繰り返し載荷させた。載荷速度は0.05Hz、雰囲気温度は常温、1473K、1673Kでそれぞれ行った。

2. 2 レーザー顕微鏡による観察

高温での耐火物の相の直接的な観察はレーザー顕微鏡を用いて行った。れんがサンプルを100 μ m以下に粉碎し、この粉状サンプル0.1gを、白金板を底部に敷いたアルミナ坩堝(ϕ 8-5mm)に設置した。加熱ステージにアルミナ坩堝をセットした後、加熱中の状況を観察した。加熱パターンは室温から1273Kまでは1000K/minで、1273Kから1773Kまでは20K/minとした。雰囲気はN₂とした。

3. 実験結果

Fig.2に珪石れんがにおける常温、1473K、1673Kの高温での作用応力上限比と破壊までの載荷回数(関係(S-N曲線))を示す。同一作用応力比に対する疲労寿命は常温に比べると、高温では大きく増加した。

Fig.3に珪石れんがの常温での繰り返し圧縮試験(疲労圧縮試験)を行ったときの試料高さ方向の見かけの歪み量の推移を示す。見かけの歪み量(ε)は h_0, l をそれぞれ試料高さ、ストローク長さとして(1)式で定義した値を用いた。

$$\varepsilon = \frac{h_0 - l}{h_0} \dots\dots\dots (1)$$

常温での見かけの歪み量は繰り返し圧縮応力付与時ではほんのわずかに増加する傾向が見られた。一方、Fig.4には高温での繰り返し圧縮試験(疲

mineral phase composition, apparent porosity ratio, bulk density and mechanical strength of the silica brick used in this study. Cubic specimens (20mm $\times$ 20mm $\times$ 20mm) were used. A schematic diagram of the experimental procedure is shown in Fig.1. Cyclical compressive loading was applied to the specimens through the cylinder of the testing machine using the waveform shown in this figure. Compressive strength, defined as the static modulus of rupture under compressive loading, was measured in each brick at room temperature and high temperature before the fatigue failure test. Table 1 also shows the compressive strength of each brick at the experimental temperatures, which were 298K (room temperature), 1473K and 1673K. Compressive strength was evaluated by the following method. A static compressive load was given to each sample at a constant loading speed (0.5mm/min), and compressive strength was obtained by calculation by dividing the load when the sample was crushed by the loading area. Fatigue failure tests were conducted by applying cyclical compressive loading under an air atmosphere. The lower limit of compressive loading was a constant value of 10% of compressive strength (σ_c), while the upper limit of loading was changed from 70% to 100% of compressive strength (σ_c) with the loading frequency of 0.05Hz. The ambient temperature was room temperature (298K), 1473K or 1673K.

2. 2 Observation by laser-scanning microscope

Direct observation of the high temperature phases in the refractory was performed by using a laser-scanning microscope. The sample brick was crushed to under 100 μ m, and a powdered sample of 0.1g was inserted into an alumina crucible in which a Pt plate was laid on the bottom. The size of the alumina crucible was ϕ 8mm-5mm. After the crucible was set

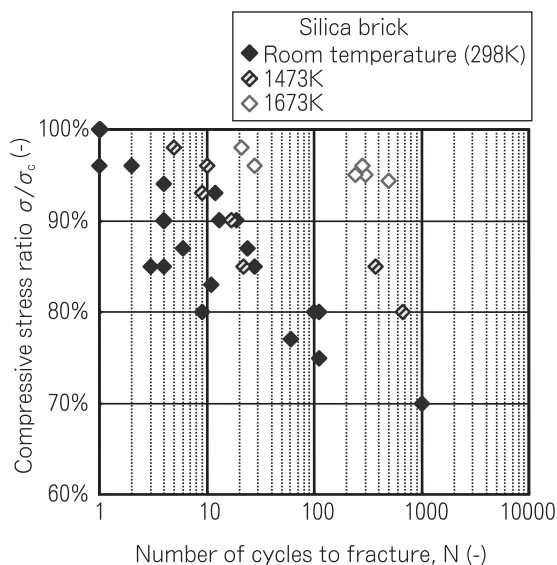


Fig.2 Relationship between compressive stress ratio and number of cycles to fracture for silica brick at room temperature, 1473K and 1673K.

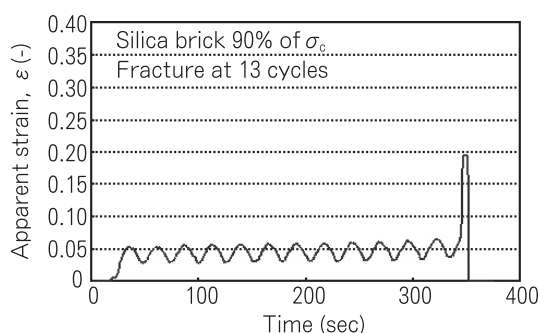


Fig.3 Trend of apparent strain in vertical direction of sample during fatigue failure test at room temperature.

労圧縮試験)を行なったときの試料高さ方向の見かけの歪み量の推移を示す。高温での見かけの歪み量は繰り返し圧縮応力付与時では大きく増加する傾向が見られた。この結果から高温での繰り返し応力負荷時には耐火物中に亀裂進展以外に一部クリープ変形が起こっている可能性があると考えられる。

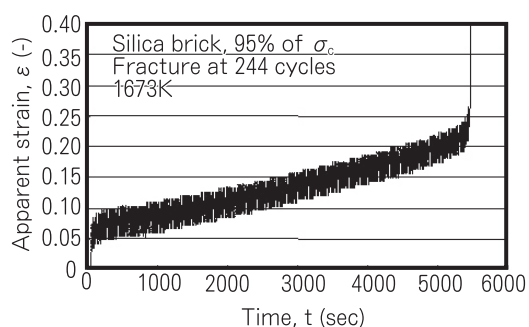


Fig.4 Trend of apparent strain in vertical direction of sample during fatigue failure test at high temperature.

in the heating stage, the sample was observed during heating. The temperature elevating rate was set to 1000K/min from room temperature to 1273K and to 20K/min from 1273K to 1773K. The atmosphere in the stage was nitrogen.

3. Experimental Results

Fig.2 shows the relationship between the compressive stress ratio (σ/σ_c) and the number of repetitions, that is, the S-N curve, at room temperature and high temperature. From this figure, it is obvious that the life of the sample becomes significantly longer with increasing temperature. Fig.3 shows the trend of apparent strain in the vertical direction of the sample in the room temperature fatigue failure test. Apparent strain ε was defined as shown in the following equation, where h_0 is sample height and l is stroke length.

$$\varepsilon = \frac{h_0 - l}{h_0} \dots\dots\dots (1)$$

Apparent strain increased slightly in the silica brick. Fig.4 shows the same trend at the high temperature, indicating a large increase under the cyclical compressive stress. This result suggests the possibility that, in addition to crack generation or crack growth, partial creep deformation takes place inside the brick at high

4. 考察

4.1 破壊力学的検討

上記に示すS-N曲線から、各種条件における珪石れんがの破壊挙動を比較するために破壊力学的検討を行った^{17),18)}。破壊後の試料を観察したところ、亀裂はほぼ荷重印加面に対して縦方向(高さ方向)に進展していた。また、図示していないが、繰り返し荷重付与時の動的弾性率は、徐々に低下した。これらの結果から、Irwinの線形理論を適用すると、材料の破壊モードはモードIとみなされる¹⁹⁾。また、材料内に存在する亀裂は繰り返し荷重付与によって徐々に進展すると考え、亀裂進展挙動にパリス則が適用できると仮定した。このとき材料内の亀裂進展速度は(2)式で表される^{17),18)}。

$$\frac{da}{dN} = C \cdot \left(\frac{\Delta K}{K_{IC}} \right)^n \dots\dots\dots (2)$$

ここで、 a は亀裂長さ、 N は繰り返し载荷回数、 K_{IC} は破壊靱性値、 C および n は固有指数である。 ΔK は最大応力、最小応力印加時の応力拡大係数の差であり、(3)式で示される。

$$\Delta K = K_{\max} - K_{\min} \dots\dots\dots (3)$$

なお、 $K_{\max}$ 、 $K_{\min}$ は最大応力、最小応力、形状因子 Y 、クラック長さ、最大、最小荷重時の応力 $\sigma_{\max}$ 、 $\sigma_{\min}$ を用いて(4)式で表される。

$$K_{\max} = Y \sigma_{\max} \sqrt{\pi \cdot a} \quad K_{\min} = Y \sigma_{\min} \sqrt{\pi \cdot a} \quad \dots (4)$$

(4)式を(3)式に代入し、さらにこの(3)式を(2)式に代入して積分すると、最終的に(5)式が導かれる。

$$\log \Delta \sigma = -\frac{1}{n} \log N + \frac{1}{n} \log \left[\frac{2K_{IC}^n}{C \cdot Y^n \cdot \pi^{\frac{n}{2}} \cdot \Delta \sigma^n (n-2)} \left[a_0^{-\frac{n-2}{2}} - a_c^{-\frac{n-2}{2}} \right] \right] \quad (5)$$

この(5)式において、各種疲労試験における最大、最小応力差 $\Delta \sigma$ 、および破壊までの回数 N をプロットし、直線関係を仮定すると、その傾きから指数 n が求められる。 n の値は材料の構成、温度、試験環境などにより変化し、物理的には亀裂の進展や応力拡大係数の変化に対する敏感性の度合

temperatures over 1473K.

4. Discussion

4.1 Fracture dynamical discussion of refractory material

Based on the observed relationship between σ/σ_c and N , the fatigue fracture behavior of the silica brick was examined using fracture mechanics in order to compare the fracture behaviors under various conditions^{17),18)}. The samples which were examined after fatigue fracture showed that cracks had mainly progressed in the direction perpendicular to the loading phase. Although not shown in a figure, the dynamic elastic modulus during the fatigue failure test was found to decrease gradually. From these results, the fracture mode was regarded as mode I, according to Irwin's linear elasticity theory¹⁹⁾. It was assumed that an existing crack will progress gradually and in a stable manner under cyclical compressive loading, and Paris's law can be applied to the crack progress behavior. The rate of crack growth can be expressed by Eq. (2)^{17),18)}.

$$\frac{da}{dN} = C \cdot \left(\frac{\Delta K}{K_{IC}} \right)^n \dots\dots\dots (2)$$

where, a is crack length, N is number of cycles, K_{IC} is fracture toughness, C and n are characteristic constants dependent on the material and ΔK is the difference of the stress intensity factor expressed by Eq. (3).

$$\Delta K = K_{\max} - K_{\min} \dots\dots\dots (3)$$

where, $K_{\max}$ and $K_{\min}$ are expressed as shown in Eq. (4), which was determined from the crack length and dimensions of the material, a geometrical factor Y and the maximum and minimum applied stresses, $\sigma_{\max}$ and $\sigma_{\min}$.

$$K_{\max} = Y \sigma_{\max} \sqrt{\pi \cdot a} \quad K_{\min} = Y \sigma_{\min} \sqrt{\pi \cdot a} \quad \dots (4)$$

いを意味し、動的熱疲労破壊抵抗係数の検討結果より、 n 値が小さいほど疲労強度の低下が著しいと考える事が出来る⁵⁾。よって、各条件において、指数 n を比較した。各種条件における指数 n 値の一覧を Table 2 に示す。高温で n 値は大きく向上し、特に 1673K では常温の 5 倍の値になった。

4. 2 高温でのれんがの疲労破壊挙動

Fig.5 に高温レーザー顕微鏡を用いて測定した珪石れんがの粒子群の写真を示す。常温から

Table 2 Values of index n at test temperatures

Temperature	n
298K	20.0
1473K	26.7
1673K	105.3

By substituting Eq. (4) into Eq. (3), substituting the result into Eq. (2) and further integrating the result of substitution, Eq. (5) was finally obtained.

$$\log \Delta\sigma = -\frac{1}{n} \log N + \frac{1}{n} \log \left[\frac{2K_{IC}^n}{C \cdot Y^n \cdot \pi^{\frac{n}{2}} \cdot \Delta\sigma^n (n-2)} \left[a_0^{-\frac{n-2}{2}} - a_c^{-\frac{n-2}{2}} \right] \right] \quad (5)$$

When the results of the fatigue failure test, that is to say, the difference of the maximum and minimum stresses, $\Delta\sigma$, and the number of cycles to rupture, N , were substituted in Eq. (5), and a linear relationship between $\Delta\sigma$ and N was assumed, the numerical constant n could be obtained.

It is considered that the numerical index n

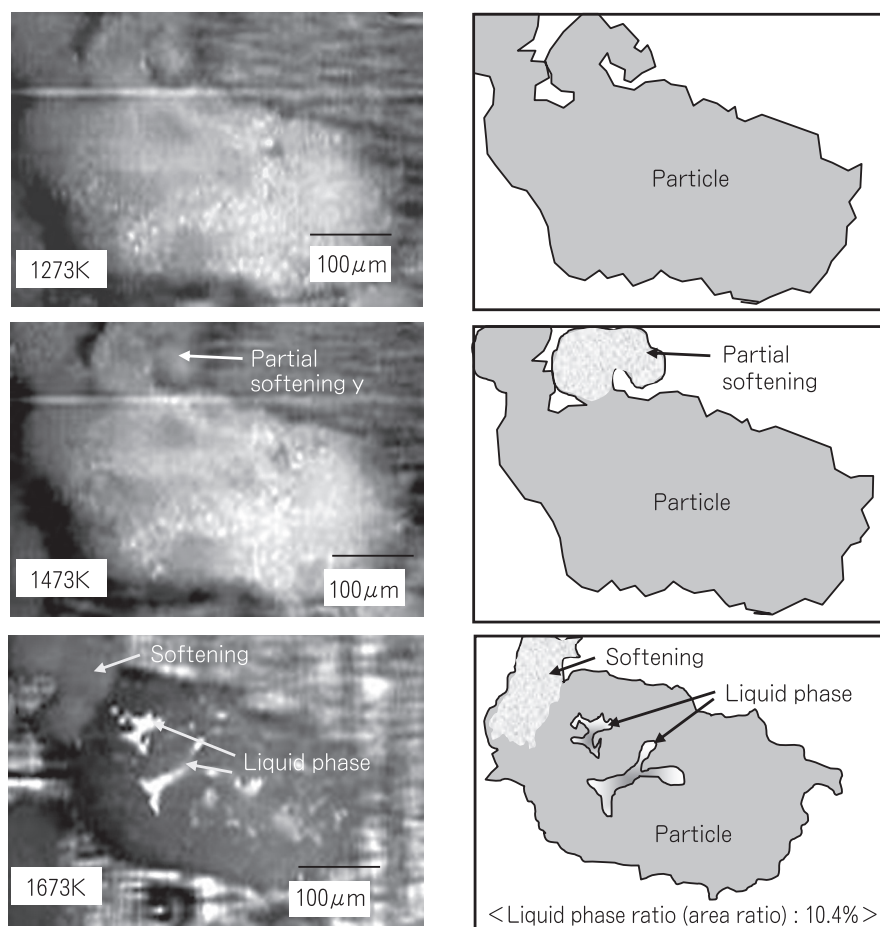


Fig.5 Photographs and schematic images of particles (pieces of fragment of brick) of silica brick observed by using laser-scanning microscope at high temperature.

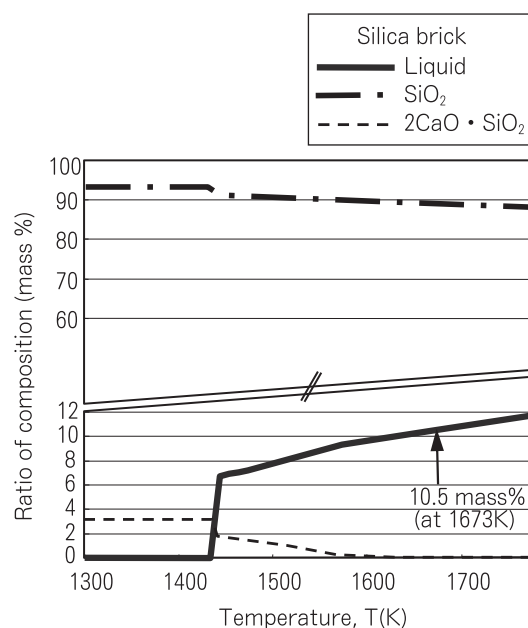


Fig.6 Calculated equilibrium phase component obtained by thermodynamic simulation based on chemical composition of bricks.

1273Kまでは粒子群に変化は見られなかったが、1473K付近より粒子群外側の一部が軟化する様子が確認され、さらに1673Kでは粒子群の内部に少ないながらも液相が観察された。

Fig.6には珪石れんがの化学組成を元に熱力学計算 (FACTSAGEによる計算) により平衡相を算出した結果をそれぞれ示す。実際の耐火物は様々なサイズの粒子群の不均質な分布によって構成される。特に、CaOやMgOなどの不純物が偏析している可能性もあり得る。しかしながら、以下に示す事実より、今回は平衡状態化学組成に関しては平衡状態を仮定した。

- ・ れんがの化学組成をランダムに分析したところ、ほぼ同等の組成であった。
- ・ 高温でのXRDの回折パターンより、析出相が平衡計算で求められた固相と一致した。
- ・ Fig.5より、面積比率がmass%に換算できると考え、面積比率から求めた1673Kでの液相率が10.4%であり、これがFig.6による平衡計算値10.5%とよく一致した。

珪石れんがでは、1458K以上で液相が生成すると計算された。それぞれのれんがの液相率は、6.7

depends on factors such as the composition of the material, the experimental temperature and the test environment and situation. The physical implication of the index n is the degree of sensitivity to changes in the progress of the crack and in the stress intensity factor. It has been reported that smaller values of n indicate a decrease in fatigue strength.⁵⁾ In this study, the values of n were compared for each experimental condition. Table 2 shows the values of n obtained from the results of the fatigue failure test of the silica brick at the various test temperatures. The values of n of the silica brick showed large increases at high temperature. In particular, at 1673K, the n value of the silica brick was about five times larger than that at room temperature.

4. 2 Fatigue fracture behavior of refractories at high temperature

Fig.5 shows photographs of particles of the silica brick (fragments of the brick) taken with a laser-scanning microscope at high temperature. The particle shape did not change from room temperature to 1273K, but at temperatures over 1273K, softening occurred from the particle edge, and at 1673K, a slight amount of liquid phase partially formed.

Fig.6 shows the calculated equilibrium phase composition as obtained by thermodynamic simulation (calculations by FACTSAGE) based on the chemical composition of the brick. Actual bricks consist of particles of various sizes with a probable heterogeneous distribution. In particular, segregation of minor components such as lime or periclase is a possibility. However, based on the following facts, the equilibrium state can be assumed in terms of the chemical composition.

- ・ The chemical composition of each brick was analyzed after random sampling. Analyses of several portions proved that the compositions

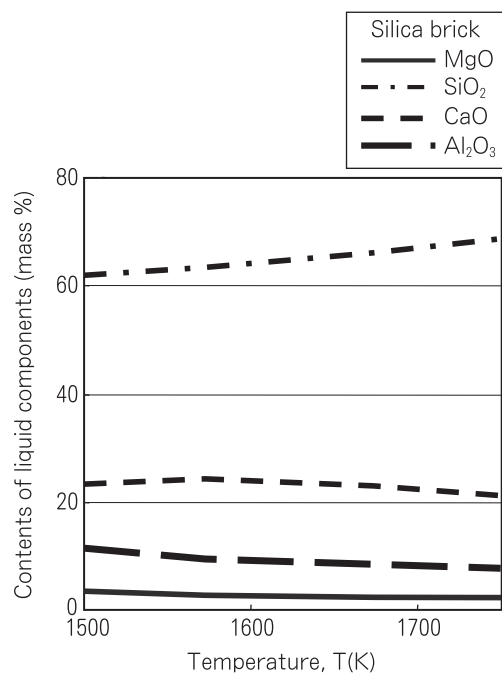


Fig.7 Calculated composition of liquid phase in each brick at high temperature obtained by thermodynamic simulation.

Table 3 Contact angle between a SiO₂ solid and a SiO₂-rich liquid

Liquid	Solid	Contact angle (deg.)	Adhesive force (N/m)
SiO ₂ rich phase	SiO ₂	140-150°	0.05-0.09 N/m

～11.7%程度であった。このことから、高温で生成した微量な液相が、本研究にて測定した耐火物の高温での疲労特性向上に寄与したものと考えられる。さらに、熱力学計算より求めた珪石れんがの高温で生成した液相の組成をFig.7に示す。珪石れんがではSiO₂が60%以上の融体と計算された。

ここで、SiO₂系融体とSiO₂固体との界面物性のデータ一覧をTable 3に示す。SiO₂系融体とSiO₂固体との接触角は140～150deg.であり、濡れ性が悪いと報告されている^{(20),(21)}。以上までの検討結果から、珪石れんがにおける高温での疲労寿命向上要因機構について、定性的ではあるが、以下のよう推定した。

were substantially the same in each random portion.

- High temperature XRD identified the same solid phases as those calculated by assuming equilibrium.
- The liquid phase ratio of 10.4 area% at 1673K, visually measured from Fig.5, was in good agreement with the calculated value of 10.5 mass% realized in Fig.6, considering the fact that the area% is convertible to mass%.

A liquid phase of as much as 10% was generated at temperatures over 1458K. The calculated liquid phase ratio was 6.7-11.7% in the silica brick. This small amount of liquid phase which is generated in the bricks at high temperature may play an important role in improved high temperature fatigue fracture life. Fig.7 shows the calculated composition of the liquid phase in the brick at high temperature. In the silica brick, the liquid phase was calculated as being a SiO₂ rich melt with a SiO₂ content over 60%. Table 3 shows the contact angles between a SiO₂ solid and a SiO₂-rich liquid^{(20),(21)}. It has been reported that the contact angle between a SiO₂ rich liquid and SiO₂ solid is 140-150°, implying poor wettability.

From the results of the above discussion, the mechanism of improved high temperature fatigue fracture life in the silica brick was deduced to be as follows. Acknowledging the qualitative nature of these conjectures, Fig.8 shows a schematic diagram of the behavior of the refractory particles at high temperature when cyclical compressive stress is applied to the silica bricks having poor wettability between liquid and solid phase. A very small amount of liquid phase with a sufficient thickness remains between the aggregate and the matrix. Due to its poor wettability, this liquid phase takes a spherical shape, allowing the liquid phase to absorb stress (by analogy, functioning like

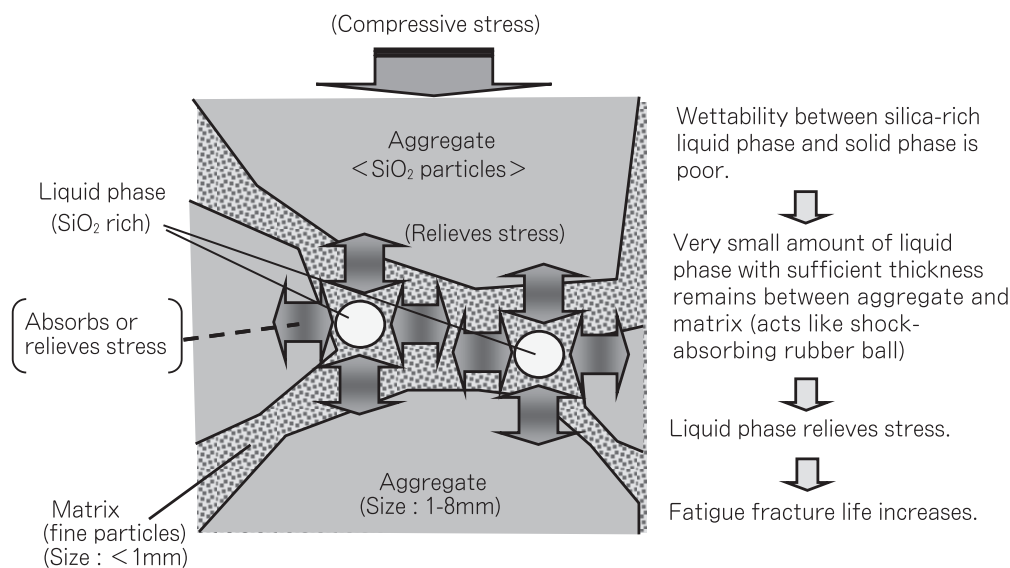


Fig.8 Schematic diagram of high temperature behavior of silica brick.

珪石れんがにおける高温での繰返し応力印加時の挙動の模式図をFig.8に示す。SiO₂含有量の多い珪石れんがでは、高温時での圧縮応力印加時にSiO₂ Richな液相が骨材/マトリックス間に存在する。この生成したSiO₂ Richな液相はSiO₂固体間の濡れ性が悪いため、粒子間に球形のような形状で存在すると考えられる。その液相が圧縮応力を緩和する、いわゆるクッションの役割をなしたため、固体/個体間で亀裂の進展が抑制され、高温での疲労寿命が大きく向上したと推定される。

しかしながら、高温における耐火物疲労破壊挙動についてはまだ疑問点も多く、今後はさらに高温での疲労メカニズムの解明、モデル化を行っていく必要がある。

5. 結言

珪石れんがの常温、高温における繰返し疲労挙動を調査した。得られた知見を以下に示す。

- 1) 1473K以上の高温での珪石れんがの繰返し疲労寿命は常温に比べ遥かに高くなった。
- 2) 高温における珪石れんがの歪み速度は、亀裂進展により進行するだけでなく、クリープ変形を伴いながら進行していくと考えられる。
- 3) 熱力学的検討より、少量の液相が高温で生成

shock-absorbing rubber balls). Thus, when compressive stress is applied, this liquid phase relieves that stress, resulting in longer fatigue fracture life.

Nevertheless, many questions about the high temperature fatigue fracture behavior of refractory materials remain to be answered. In the future, it will be necessary to clarify the mechanism of fatigue failure and analyze fatigue fracture behavior at high temperature.

5. Summary

The fatigue failure behavior of silica brick was investigated at room temperature and high temperature. The results are summarized as follows.

- 1) The fatigue failure life of silica brick showed large increases at 1473K and higher temperatures.
- 2) At high temperature, it is thought that the strain rate in the silica brick increases not only as a result of crack growth, but also as a result of creep deformation.
- 3) A thermodynamics simulation showed that a small amount of a liquid phase is

することが示された。このことより、高温での耐火物の繰り返し疲労特性は、微量液相の生成が関与し、固相/液相の濡れ性の良し悪しにより疲労寿命の向上度合いが変化すると推定される。

generated in the brick above a certain temperature. It was deduced that the fatigue fracture behavior of the brick at higher temperatures is related to the formation of this liquid phase. Based on calculated differences in wettability, a mechanism in which the liquid phase performs a stress relieving function was proposed as an explanation of the improved fatigue failure life of silica brick at elevated temperatures.

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