Creep Failure Process for Fine Powder Beds by Tensile Loading †

Hidehiro Kamiya***, Makio Naito**,
Jun-ichiro Tsubaki***** and Genji Jimbo****
Department of Chemical Engineering, Nagoya University*

Abstract

To investigate the process of creep failure phenomena in fine powder beds, the change in the tensile failure strength and the displacement of powder beds during the creep failure process was analyzed. The increasing displacement is divided into three sections (I, II, III). The increasing rate of displacement slows in the first stage (section I), remains almost constant in section II, and rapidly increases and falls in section III. By using the experimental equation to relate the creep failure life to the increasing rate of displacement in section II, the change in tensile strength of powder beds can be estimated during the creep failure process. No decrease in tensile strength is observed during the first stage (section I). However, in the second and third stages (section II and III), the strength decreased remarkably with increasing creep time. The increasing displacement can be fitted by a Voigt model in the region of section I, in which the tensile strength is almost constant. According to the elastic/viscous parameters of a Voigt model decrease in relation to the decrease in fracture strength of powder beds, the complex increase of displacement in the region of sections II and III can be quantitatively estimated. The deformation resistance of powder beds was quantitatively related to the fracture strength.

1. Introduction

Because fine powder of less than 10 μm in primary particle size, due to a high adhesiveness, causes such problems as blockage, measurements and analyses of adhesive force of fine particles have been conventionally conducted for primary particles²⁾ or powder beds¹⁶⁾. In addition, with fine particles of high adhesiveness, such time-dependent phenomena as fatigue, creep and stress relaxation are significantly observed even in a dry powder bed. Such phenomena cannot be explained simply by the adhesive force alone, and need to be analyzed by considering the time-dependence change of micro structure in powder beds constituting the fine powder. In the field of soil engineering, the time-dependent phenomena have been conventionally addressed to in the setting of wet particle beds^{3,9)}, and studied as a continuum by means of various viscoelasto-plastic models^{1,17,21)} and using

models with microscopic changes in the configuration of particles taken into consideration^{11,12)}. On the other hand, it is reported that a dry fine powder bed is also highly densified by repetitive consolidation and creep consolidation^{4,7,8)} in relation to a ceramics forming operation. Naito, et al.^{13,14,20)} pointed out that fatigue and creep failure were caused also by tensile loading and shearing force experimentally by repetitively wiping off a collected powdery dust bed, and confirmed the presence of such phenomena using a fundamental experiment^{5,6)}. Although a macroscopic behavior of deformation to failure of a dry fine powder bed is similar to that of a wet bed of relatively large particles, for wet particles, interstitial water in gaps between particles significantly affects the time-dependent phenomena. In the case of dry fine particles, the time-dependent phenomena are considered to be caused by gradual deformation in such secondary particle structure as an aggregation of particles, and although the macroscopic behavior is similar to that of wet particles, the driving mechanism is supposed to be different.

Such similar phenomenon is reported whereby the ratio of removal and/or dispersion is increased with an increase in the number of repetitions, when a removal force is repetitively applied to a powder bed for the removal of particles collected onto a substrate¹⁵⁾

* Furo-cho, Chigusa-ku, Nagoya 464-01 JAPAN

** Now with Fine Ceramics Center, Nagoya 456

*** Now with Tokyo University of Agriculture and Technology

**** Now with Chubu Powtech Plaza Laboratory

***** Now with Department of Micro-system Engineering Graduate school of, Nagoya University

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and dispersion of particles from the surface of a particle bed⁹⁾. It has been attempted to explain such phenomenon as a probable phenomenon of failure due to fluid-dynamic unevenness of the removal force. However, it can be considered based on a theoretical phenomenon of failure that the configurational structure in a powder bed is gradually changed by the action of a repetitive force, and a final failure is caused because the resistance against failure stress is reduced. However, such failure mechanism is rarely taken into consideration, and the process leading to failure has rarely been determined and examined in detail.

Accordingly, we measured the rate of displacement and change in failure stress during a process leading to a creep tensile failure in two kinds of inorganic fine powder beds, and considered the changes in the dynamic characteristics of a powder bed in a process leading to creep failure. Then, we devised a viscoelasto-plastic model for commonly expressing changes of failure stress and behavior of deformation.

2. Experiments

For the experiments, a suspended-type adhesion measurement apparatus (Hosokawa Micron Co., Ltd., Cohetester) shown in **Figure 1** was employed. The apparatus is a two-piece tensile failure tester of vertical consolidation and horizontal tension type, as described in detail in our previous report. It allows testing at a constant stress loading rate by employing a spring of low modulus of elasticity between a movable cell and a pulley which is rotated at a constant speed to load the cell. The density and mean particle size of powder samples used in the experiments are comprehensively shown in **Table 1**. Sample I is a standard powder sample (Kanto Loam JIS Classification 11), and Sample II is a fused alumina powder (Showa Denko K.K., WA #8000) used as an abrasive. Both are fine powders of about 1 μm in mean particle size.

The powder samples were loaded onto the measurement cell in an atmosphere of constant temperature and humidity ($20^\circ\text{C} \pm 5^\circ\text{C}$, $60\% \pm 10\%$), and subjected to a preliminary loading of 5 kPa for 10 min to achieve an approximately constant porosity of 0.75 and 0.74 for Samples I and II, respectively. Three

Fig. 1 Scheme of the test apparatus for measurement of tensile strength of powder beds.

types of tensile failure tests outlined in **Figure 2** were conducted on the powder beds. **Figure 2 (a)** shows a directional tensile displacement of the powder beds during the experiments, and **Figure 2 (b)** a time-course change of loading stress. Experiment 1 is a simple tensile failure test for measuring the tensile stress (σ_z) and determining a relation between the stress and displacement to failure by increasing the tensile stress at a constant rate. Experiment 2 is a creep failure test for determining time (t_b) to creep failure by increasing the tensile stress to a specified stress (σ_s) equal to or lower than the mean tensile failure stress of the powder bed obtained in Experiment 1, then, maintaining the stress constant, and measuring the increasing displacement as shown in **Figure 2 (a)**. The increasing displacement to creep tensile failure is divided into three sections, Section I or an early stage showing a low increasing rate of displacement, Section II with an almost constant increasing rate and linear time-course increase of displacement and Section III showing a rapid increase of displacement leading to creep failure^{6, 10)}. It was also confirmed for creep or fatigue failure of such ductile material as metals and in the soil-dynamic field that the increasing displacement in creep and other time-dependent failure can be divided into three sections. This was also observed in a high frequency repetitive stress fatigue test of dry fine powders. Presently, no model is proposed for commonly evaluating the complex increase of displacement. We studied a relation between life to creep failure (t_b) and time (t_I , t_{II}) from Section I to Section II and Section II to Section III, and determined a method for estimating the life to creep failure from the behavior of deformation before failure. In Experiment 3, a tensile stress (σ_E) was obtained by increasing the tensile stress again at a predetermined time (t_C) during the creep failure test. From the result, the change in strength of the powder bed during the process to creep failure was evaluated. The specified creep stress (σ_s) and time for starting the measurement of the strength were determined from the results of Experiments 1 and 2.

Table 1 Powder properties of each sample.

Sample name		Mean diameter [μm]	True density [kg/m^3]
Sample I	Kanto loam (JIS No.11)	2.0	3.0×10^3
	Fused alumina (WA #8000)	1.3	3.9×10^3

Fig. 2 Changing process of the displacement of powder beds and loading stress during simple tensile failure test (test 1), creep failure test (test II) and tensile failure test during creep failure process.
(a) Increasing process of displacement during each test.
(b) Changing process of tensile stress during each test.

3. Results

3.1 Tensile failure test (Experiment 1)

The result for Sample I is shown in **Figure 3** as an example of the relation between stress and displacement obtained by a simple tensile failure test of powder beds. The stress (σ_z) at failure widely varied, and the coefficient of variation of the tensile stress at failure was 2.67% for 10 samples measured. However, the relation between stress and displacement shows a relatively similar curve, the displacement at failure is almost at the same point, and it is observed that the samples tend to break when the displacement reaches a certain value. Thus, in **Figure 3** (b), the loading stress and displacement in the ordinate and abscissa are changed into dimensionless values by means of the stress (σ_z) and displacement (Δ_z) at failure, and shown as a ratio of loading stress (σ/σ_z) and dimensionless displacement (Δ/Δ_z). In terms of the ratio of loading stress, a significant coincidence is observed in the relation between stress and displacement, and the coefficient of variation is reduced to 1.25% for a variation of displacement at $\sigma/\sigma_z = 0.95$. A similar result is obtained with Sample II, and it

Fig. 3 Examples of tensile stress and displacement relations during simple tensile failure test (test 1).

is considered that a simple tensile failure of powder beds is a phenomenon attributed to displacement such that failure occurs as the displacement reaches a certain value, and the variation is reduced when it is determined from the aspect of displacement. Accordingly, an average curve of the ratio of stress against the displacement is obtained, as shown in **Figure 3** (b), and defined by a function f .

$$\sigma/\sigma_z^* = f(\Delta/\Delta_z) \quad (1)$$

In the creep tensile test, the stress is increased to a specified stress (σ_s) lower than the tensile failure strength (σ_z) before starting the creep test. From a displacement (Δ_0) at the beginning of the creep test and Eq. 1, an estimated tensile failure strength (σ_z^*)

of a powder bed subjected to the creep test can be obtained by the following equation.

$$\sigma_z^* = \sigma_z / f(\Delta / \Delta_0) \quad (2)$$

Life to creep failure and the change in strength of a powder bed during the creep test are not sufficiently high compared to the variation in the tensile failure strength of the powder bed without such hysteresis. Therefore, it is required to estimate the tensile failure strength of the powder beds that are subjected to the creep test and the like to evaluate the change in the strength.

3.2 Creep tensile failure test (Experiment 2)

A relation between the loading strength and life obtained by the creep test is shown in **Figure 4**. In the ordinate, the specified stress (σ_s) is changed into a dimensionless value by means of the estimated tensile failure strength (σ_z^*) that is obtained by Eq. 2. By estimating the tensile failure stress, and obtaining the ratio of loading stress, a variation of life at the same value in the ordinate can be reduced. From the figure, if the ratio of loading stress is set to 0.9 or less, n_o failure occurs within 10^4 s, which is the test range specified for the study. Because a fine powder bed may be stabilized, and have a higher strength, if no failure occurs due to a tensile force for 10^4 s or a longer period¹³⁾, the measurement of the strength during the process to creep failure (Experiment 3) is conducted at $\sigma_s / \sigma_z^* = 0.95$ to ensure the occurrence of a creep failure within 10^4 s.

The increasing displacement before creep failure can be divided into three sections, as shown in **Figure 2 (a)**. The result of plotting the deformation rate (C_{II}) against life to creep failure during Section II of the process, in which the displacement increasing rate remains almost constant, is shown in **Figure 5**. Both Samples I and II are substantially approximated to a straight line of slope -1 . Thus, if Section II is confirmed, the life to creep failure can be estimated in a non-destructive way from the deformation ratio

Fig. 4 Stress and creep failure life relations of sample 1.

Fig. 5 Relations between creep failure life and constant deformation velocity of powder beds during section II.

(C_{II}) by means of the following equation.

$$C_{II} = -6t_b^{-1} \quad (3)$$

A similar relation is reported for a repetitive stress fatigue test of a fine powder bed⁵⁾ and a creep failure test of wet clay¹⁸⁾. As an alternative method for estimating the life from the behavior of deformation, times (t_I , t_{II}) from Sections I to II and II to II can be employed. In **Figure 6**, a relation between (t_I , t_{II}) and life values is shown. In both Samples I and II, it is observed that t_b and t_I , t_{II} are proportional, and the ratio of Sections before creep failure is almost constant. In addition, t_{II} is almost unchanged between two types of samples, which means the occupying ratio of Section III is almost identical.

Fig. 6 Relations between creep failure life and the ending time (t_I , t_{II}) of section I and II.

3.3 Strength measurement test during the process to creep failure (Experiment 3)

The result of Experiment 1 indicates that the effect of variation in the tensile strength on the life to creep failure can be reduced in a creep test by maintaining the displacement (Δ_0) constant during the simple tensile test before the creep test. From Experiment 2, it was found that the life to failure can be estimated in a non-destructive manner from the rate of deformation, if the onset of the constant rate deformation, section II, can be confirmed in a creep test. On the basis of such results, an experiment for measuring the strength during the process to creep failure was conducted, and changes in strength were determined. In **Figures 7, 8**, the results of strength measurement at $\sigma_S/\sigma_Z^* = 0.95$ during a process to creep failure are shown respectively for Sections I, II and III. With regard to the strength measurement data in Sections II and III, the life value (t_b^*) was estimated by means of Eq. 3, and the ratio to start time (t_c) of the strength measurement test was obtained for the abscissa. The ordinate shows the strength measurements with the hysteresis of creep tension changed into a dimensionless value by means of the estimated strength when no hysteresis is applied. From the result of **Figure 6**, the ratios of Sections I, II and III remain unchanged between samples. Since the data of strength measurements during Section I is not useful for life estimation, it is comprehensively shown at 0.2 of (t_c/t_b^*) in the abscissa.

Fig. 7 Change of tensile strength during creep failure process. (Sample 1)

Fig. 8 Change of tensile strength during creep failure process. (Sample 2)

The strengths measured during Section I are distributed around 1.0 of the ordinate, and no reduction in strength is observed. Therefore, it is considered that Section I does not represent a deformation accompanied by a reduction in strength but a deformation for balancing and stabilizing the structure of the powder bed against creep tensile stress. In Section II, the value of the ordinate exceeds 1.0 in very few data, which means that the strength is lower than that of Section I. In Section III, the strength is further reduced approximately to the specified stress. In **Figure 8** showing the result for Sample 2, a similar result is obtained. Accordingly, it can be considered that reduction in strength to creep failure of a powder bed does not gradually occur through an entire process. Almost no reduction occurs during Section I, the strength begins to decrease in Section II, and creep failure occurs as soon as $s_E = s_S$ is reached. As a result, it is expected that some correlation may be present between the change in strength and the complex increasing displacement to creep failure.

4. Discussion

The reduction in strength and behavior of deformation during a process to creep failure is now quantitatively evaluated by means of a Voigt model in **Figure 9 (a)**. For the Voigt model, a relation between stress

Fig. 9 Rheological model to analyze the increasing process of displacement.
(a) Voigt model
(b) Scheme of increasing process of displacement described by a Voigt model

and displacement is expressed by the fundamental equation shown below.

$$\sigma = k_p \Delta + \mu d\Delta/dt \quad (4)$$

where k_p is the modulus of elasticity of the spring element, and μ the coefficient of viscosity of a dash pot element. By solving the equation at $\sigma = \sigma_s =$ constant as in the creep test, a time-course change of displacement Δ can be expressed by the following equation.

$$\Delta(t) = \sigma_s/k_p \{1 - \exp(-k_p t/\mu)\} \quad (5)$$

The equation represents a process of deformation at a rate gradually decreasing to a constant value. For a specified tensile stress ($\sigma_s/\sigma_z < 0.9$), an increasing displacement is sufficiently approximated by the Eq. 5, and the rate of deformation is found to be about 0 after a creep time of $10^4 s$. Even in a condition causing creep failure, the deformation process can be sufficiently approximated within the range of Section I. It is very difficult to express the complex process of deformation through Sections II and III to creep failure by a single viscoelasto-plastic model.

Therefore, it is assumed in this paper that the viscoelastic parameters (k_p , μ) change together with the tensile stress of a powder bed. Such assumption is made in the field of soil dynamics for the study of wet particles whereby a viscoelastic parameter indicative of a deformation resistance is a function of failure criterion determining the strength^{1, 17, 21)}. Here, it is assumed that the viscoelastic parameters are constant (k_{pI} , k_{pII}) in Section I, but decrease progressively in Sections II and III. A change in tensile displacement (Δ) in Sections II and III is approximated by the following equations.

$$\text{(Section II)} \quad \Delta(t) = C_{II}(t - t_I) + \Delta_I \quad (6)$$

(Section III)

$$\Delta(t) = C_{III}(t - t_{II})^2 + C_{II}(t - t_I) + \Delta_I \quad (7)$$

where Δ_I is a cumulative displacement at the end of Section I, and C_{II} , C_{III} are constants. By substitution of Eq. 6 and Eq. 7 in Eq. 4, respectively, assuming k_p and μ are in such relation as shown in Eq. 8⁴⁾:

$$\mu(t)/k_p(t) = C = \text{constant} \quad (8)$$

and solving for $k_p(t)$, a time-course change of the elastic parameter (k_p) that represents the behavior of deformation in Sections II and III can be expressed by the following equations.

(Section II)

$$k_p(t) = k_{pI} \sigma_s / [C \{ \exp(-C t_I) \} (t - t_I) + 1] \quad (9)$$

(Section III)

$$k_p(t) = k_{pI} \sigma_s C / [k_{pI} C_{II} C(t - t_{II})^2 + C \sigma_s \{ \exp(-C t_I) \} (t - t_I) + \Delta_I] \quad (10)$$

Figure 10 shows the result of obtaining a time-course change of the elastic parameter (k_p) by using

Fig. 10 Examples of fitted and calculated results of increasing and changing process of displacement and elastic parameter in Voigt model.

Eqs. 6 to 10, and approximating the creep deformation process. The creep deformation process is sufficiently approximated by Eqs. 5 to 7. The time-course change of the elastic parameter (k_p) used for approximation is constant in Section I, and decreases in Sections II and III. The tendency of reduction of the elastic parameter (k_p) is relatively similar to the reduction of the tensile strength shown in **Figures 7 and 8**. Then, with the data of strength measurements for Sections II and III, by obtaining the value (t_c/t_b) in the abscissa of each strength measurement, and substituting it in Eqs. 9 and 10, a value of k_p corresponding to the strength measurement is obtained. The ratio (k_p/k_{pl}) of the k_p value to the elastic parameter (k_{pl}) for Section I is obtained, and a relation with the corresponding ratio of strength (σ_E/σ_Z^*) is shown in **Figures 11 and 12**. The solid line in the figures shows a proportional relation of slope 1, and the broken line the range of variation of σ_E/σ_Z^* for Section I. Between the strength and elastic parameter, a substantially proportional relation is established within a range of variation for two types of samples.

Accordingly, by assuming that the viscoelastic parameter representative of a deformation resistance is reduced proportionally with a reduction in strength due to the hysteresis of creep tensile stress, the complex behavior of deformation to creep failure can be approximated to a single viscoplasto-elasticity model. Thus, it is suggested that a close relation

Fig. 11 Relations between tensile strength and elastic parameter during creep failure process. (Sample 1)

Fig. 12 Relations between tensile strength and elastic parameter during creep failure process. (Sample 2)

exists between the strength of a fine powder bed and the deformation behavior, and that the strength and failure characteristics of a powder bed can be estimated by evaluating the displacement.

5. Conclusions

Following the measurement and analysis of the strength and deformation behavior during a process leading to creep tensile failure using two types of fine powders, the following results were obtained.

- (1) By means of a simple tensile test, it was confirmed that the tensile failure of a fine powder bed is displacement-dependent. In addition, the deformation process to creep failure is divided into three sections, and an equation (Eq. 3) for estimating the life from the rate of deformation for Section II of a constant deformation was obtained after analyzing the deformation behavior of the sections.
- (2) Although a change in strength to creep failure is substantially identical to that of a sample with no hysteresis of creep tensile stress in Section I, where the rate of deformation gradually decreases, in Sections II and III, the strength gradually decreases, and failure occurs as the strength decreases to a specified stress.
- (3) By means of a Voigt model assuming that the viscoelastic parameter decreases proportionally with a reduction in strength, the reduction in

strength and the complex process of deformation to creep failure of a powder bed was expressed in a simple manner.

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Nomenclature

C	= Ratio of elastic parameter to viscous parameter ($= kp/\mu = \text{constant}$)	[s ⁻¹]
C_V	= Coefficient of variance	[—]
C_{II}	= Mean increasing rate of displacement in section II	[m/s]
C_{III}	= Constant in Eq. (7)	[m/s ²]
$f(\Delta)$	= Function of tensile stress and displacement during simple tensile test	[—]
k_p	= Elastic parameter in Voigt model	[Pa/m]
k_{pI}	= Elastic parameter in Voigt model during section I ($= \text{constant}$)	[Pa/m]
$k_p(t)_{cal}$	= Calculated elastic parameter from Eq. (9) or (10)	[Pa/m]
t_b	= Creep failure life	[s]
t_b	= Estimated creep failure life from Eq. (3)	[s]
t_I	= Creep time at the end of section I	[s]
t_{II}	= Creep time at the end of section II	[s]
t_C	= Creep time at the start of test 3	[s]
Δ	= Displacement of powder beds	[m]
Δ_0	= Displacement of powder beds at the start of creep test	[m]
Δ_I	= Displacement of powder beds at the end of section I	[m]
ϵ	= Porosity of powder beds	[—]
σ	= Tensile stress	[Pa]
σ_E	= Tensile strength of powder beds after creep damage	[Pa]
σ_S	= Set tensile stress during creep test	[Pa]
σ_Z	= Tensile strength of powder beds determined by simple tensile fracture test	[Pa]
σ_Z	= Estimated tensile strength from Eq. 2	[Pa]
μ	= Viscous parameter in a Voigt model	[Pa s/m]

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Author's short biography

Dr. Hidehiro Kamiya

Dr. Hidehiro Kamiya is Associate Professor of Graduate School of Bio-Application & Systems Engineerings at Tokyo University of Agriculture and Technology. His major research interests are adhesion, dispersion and aggregation behavior of fine particles in water, ceramic powder processing and science, and fine ash powder behaviors in coal and solid waste (included RDF) combustion and gasifier.

Dr. Makio Naito

Dr. Makio Naito is Manager of Standardizing Project Division at JFCC. His major research interests are processing technology of fine particles and composite particle etc.

Dr. Tsubaki Jun-ichiro

Dr. Tsubaki Jun-ichiro is Professor of Department of Micro-system Engineering Graduate School of Nagoya University. His major research interests are particle characterization of ceramics powder, porous ceramic materials and ceramic process before firing.

Dr. Genji Jimbo

Dr. Genji Jimbo is the head of Chubu Powtech Plaza Laboratory and Professor of Gifu University of Economics. His major research interests are Powder Technology general.