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Study of Dynamic Behavior of Dense Silicon Nitride Ceramics at Elevated Temperature

J. Yuan and T. Shioya*

Department of Modern Mechanics, University of Science and Technology of China, Hefei 230026, P.R. China

*Department of Aeronautics and Astronautics, University of Tokyo, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113, Japan

Abstract. Impact tests of ceramics at elevated temperature up to 1400°C are performed by the split Hopkinson pressure bars with a long length specimen. A new method for determining viscoelastic mechanical property is proposed. The internal friction of dense Si_3N_4 ceramics at the frequency of 5.9 kHz and elevated temperature is evaluated. Assuming that a single thermal activation mechanism controls the deformation of the material at the range of test temperature, a two-parallel Maxwell-body model is proposed to describe the viscoelastic behaviors under axial loading. As the value of thermal activation energy, 50 kcal/mol is obtained.

Résumé: Des expériences d'impact sur céramiques ont été effectuées à haute température jusqu'à 1400°C au moyen d'une barre de Hopkinson fonctionnant en compression avec des échantillons de longueur importante. Une nouvelle méthode pour déterminer les caractéristiques viscoélastiques est proposée. La friction interne dans les céramiques denses Si_3N_4 est évaluée à la fréquence de 5.9 Hz à haute température. En supposant qu'un mécanisme thermiquement activé contrôle la déformation du matériau dans le domaine de température considéré, un modèle du type solide de Maxwell à montage parallèle est proposé pour décrire les comportements viscoélastiques sous chargement axial. On a obtenu une valeur de l'énergie d'activation thermique de 50 kcal/mol.

1. INTRODUCTION

Structural ceramics is an emerging class of engineering material with a variety of application at elevated temperature. In order to study the viscous mechanical property of silicon nitride ceramics at elevated temperature, the torsion pendulum and ultrasonic pulse measurements were performed [1,2]. The results showed that the internal friction curve of dense Si_3N_4 ceramics has a feature of peak and background components as temperatures rises under torsion loading (see Fig.1). The temperature-dependent viscous behaviors was interpreted by thermal activation mechanism. A microstructure mechanical model was established to describe its viscoelastic behaviors [3].

In the present paper, tests of dense Si_3N_4 ceramics under the axial impact loading at elevated temperature are performed by the use of split Hopkinson pressure bars (SHPB), with long length specimens adopting the circumstance of elevated temperature up to 1500 °C. Because of low internal friction of dense Si_3N_4 ceramics (see Fig.1) as well as a long length specimen a new wave propagation analysis is proposed to analyze its viscous property. The internal friction at elevated temperatures under axial impact loading is evaluated. Furthermore, the temperature-dependent internal friction curve is interpreted by the thermal activation mechanism, the thermal activation energy of dense Si_3N_4 ceramics is evaluated. A phenomenological mechanical model is established to obtain the temperature-dependent viscoelastic constitutive equation of dense Si_3N_4

Fig. 1: Internal friction of dense Si_3N_4 ceramics in torsion

ceramics at elevated temperature.

2. EXPERIMENTS AND ANALYTICAL METHOD

2.1 SHPB tests at elevated temperature and specimen

The SHPB apparatus used for the impact tests at elevated temperature is shown in Fig.2. A slender output bar is used in order to measure the small force in the output bar. The infrared image furnace provides elevated temperature circumstance up to 1500°C. It has been confirmed by the experimental measurement that the main part of specimen is in an uniform temperature distribution. The specimen material is dense Si₃N₄ ceramics. (hot-pressed sintered, with the additives of Y₂O₃, Al₂O₃ etc.) The specimen has the cross-sectional size of 4mm × 4mm with a length of 150mm. The incident, reflected and transmitted stress wave signals are recorded by the strain gauge transducers and recorders. Fig.3 shows a typical record of wave forms. Fig.4 shows the comparison of transmitted waves at different temperatures.

Fig. 2: Schematic diagram of SHPB at elevated temperature

Fig. 3: A typical record of wave forms

Fig. 4: Transmitted waves at different temperatures

2.2 Wave propagation procedure for determining internal friction

For the short length specimens, the ordinary analytical method for SHPB test is adapted to obtain the stress-strain curve of specimen material at a constant strain rate from recorded wave signals. For the long length specimens, the stress wave propagation along the specimen can not be neglected for calculating the stress-strain relationship of the specimen material. Since the analysis of wave propagation itself needs the stress-strain relationship of the specimen material, an iterative method is usually applied. However, the iterative method is difficult to apply to the present study because of the low internal friction of dense Si₃N₄ ceramics in the test temperature range. In fact, the specimen stress-strain relationship is close to the

linear elastic relation so that it is inaccurate to distinguish the viscous effect from a series of stress-strain curves. In the following, a new analytical method is proposed for determining the viscous mechanical property directly from the recorded incident and transmitted stress waves in the SHPB test.

In the typical wave propagation in the material with linear viscoelastic mechanical property, a single frequency harmonic stress wave propagates in the positive x direction, in the form of,

$$\sigma(x, t) = \sigma_0 e^{-\alpha x} e^{i\omega[t - (x/c)]} \quad (1)$$

where ω , c and α are the frequency, the velocity and the attenuation coefficient of stress wave, respectively, and σ_0 is a constant. From the measurement of c and α , the internal friction, δ (when $\delta \ll 1$), is determined according to [4], as,

$$\begin{aligned} \delta &= \alpha \lambda / \pi \\ \lambda &= 2\pi c / \omega \end{aligned} \quad (2)$$

where λ is the wave length. Hence, determining c and α from recorded incident and transmitted stress waves allows evaluating internal friction. The analysis is based on the following SHPB model for linear elastic specimen.

2.3 An elastic solution of SHPB for linear elastic specimen

Assuming the specimen is a linear elastic body and taking account of the axial inertia effect of specimen, the elastic wave propagation in the specimen of Fig.5 is described by the initial and boundary problem, i.e.,

Fig. 5: Model of SHPB for linear elastic specimen

$$\begin{aligned} \text{Controlling equation:} \quad & \rho \frac{\partial V}{\partial t} = \frac{\partial \sigma}{\partial x} \quad \frac{\partial \varepsilon}{\partial t} = \frac{\partial V}{\partial x} \quad \sigma = \rho c^2 \varepsilon \\ \text{Boundary condition:} \quad & \sigma(0, t) - \rho_0 c_0 V(0, t) = 2\sigma_I(t) \quad \sigma(L, t) + \rho_0 c_0 V(L, t) = 0 \\ \text{Initial condition:} \quad & \sigma(x, 0) = V(x, 0) = \varepsilon(x, 0) = 0 \end{aligned} \quad (3)$$

where $\rho_0 c_0$ and ρc are the density and wave velocity of bars and specimen, respectively. The σ , ε and V are the stress, the strain and the velocity of specimen, respectively. The $\sigma_I(t)$ is the incident stress wave. The Young's modulus of specimen is $E = \rho c^2$. The transmitted stress wave, $\sigma_T(t)$, can be determined by using the method of Laplace integral transforms. The final result is

$$\sigma_T(t) = \sigma(L, t) = \frac{4F}{(1+F)^2} \sum_{n=0}^{\infty} \left[\left(\frac{1-F}{1+F} \right)^{2n} \sigma_I(t - (2n+1)\tau) H(t - (2n+1)\tau) \right] \quad (4)$$

where $H(t)$ denotes the Heaviside unit function, $F = \rho_0 c_0 / \rho c$, and $\tau = L / c$. From Eqn.(4),

$$\sigma_T(t + 2\tau) = \frac{4F}{(1+F)^2} \sigma_I(t + \tau) + \left(\frac{1-F}{1+F} \right)^2 \sigma_T(t) \quad (5)$$

Substituting Eqn.(5) with

$$\begin{aligned} \sigma_T(t + 2\tau) &= 2\tau \sigma'_T(t) + \sigma_T(t) \quad (\text{when } 2\tau \ll t) \\ \sigma_I(t + 2\tau) &= 0 \quad (\text{when } t + 2\tau > T) \end{aligned}$$

where T is the time period of incident stress wave, we have

$$\sigma'_T(t) = -\frac{4F}{(1+F)^2} \frac{\sigma_T(t)}{2\tau}.$$

By solving it, we have

$$\sigma_T(t) = \sigma_0 \exp \left[-\frac{4F}{(1+F)^2} \frac{t}{2\tau} \right]. \quad (6)$$

Eqn.(6) shows that the declined part AB of transmitted wave (see Fig.3) decreases exponentially. In fact, the attenuation mechanism of AB causes from the stress wave reflection and transmission at the interfaces between specimen and bars.

Taking the different cross-section areas of specimen and bars into account, a calculation similar to the above analysis is deduced so that the transmitted stress wave is predicted from the incident stress wave. The comparison between the actual and predicted transmitted stress waves is shown in Fig.6, in which the dense Si_3N_4 ceramics shows linear elastic behavior at room temperature. Moreover, a curve fitting process of the declined part AB in Fig.6 shows that it is well expressed as an exponential function.

2.4 Determining the internal friction

Assuming the incident stress wave is a single frequency harmonic stress wave in SHPB test, the attenuation of stress wave in specimen at elevated temperature can be described by Eqn.(1). Hence, the declined part AB of transmitted wave in Fig.3 is a result of two kinds of attenuation mechanism, i.e., the attenuation of interface effect described by Eqn.(6) as well as the attenuation of specimen viscosity described by Eqn.(1). Because both attenuation mechanisms result in exponential decrement in the declined part AB of transmitted wave, AB in Fig.3 can be expressed as

$$\sigma_T(t) = \sigma_o \exp \left[-(\alpha' + \beta) \frac{t}{2\tau} \right] \quad (7)$$

where α' is the attenuation coefficient of specimen viscosity and β is the attenuation coefficient of interface effect.

The wave length is measured from the incident stress wave. The wave velocity can be determined by the same elastic stress wave propagation analysis discussed above, i.e., comparing of actual and predicted transmitted waves at different presumed wave velocities, the reliable wave velocity will result in a well fit in the early stage of transmitted waves. A more detailed discussion of such a method for determining wave velocity can be found in Ref.[5].

The values of β is determined from the predicted transmitted stress wave, meanwhile, the value of $\alpha' + \beta$ is determined from the actual transmitted stress wave. Hence, the value of α' is obtained so that the internal friction can be evaluated according to Eqn.(2).

3. INTERNAL FRICTION AND CONSTITUTIVE EQUATION

3.1 Test results

From test data the parameters of the wave velocity, the elastic modulus, the wave length and the frequency at room temperature are obtained and listed in Table 1.

The internal friction and change of wave velocity as temperature rises are shown in Fig.7 and 8, respectively. From Fig.7 it is found that the internal friction under axial loading also shows a feature of peak and background components as temperature rises.

3.2 Thermal activation process and mechanical model

The viscous behavior of dense Si_3N_4 ceramics at elevated temperature is due to the softening effect of its microstructure components. The composite microstructure of dense Si_3N_4 ceramics consists a grain phase

Fig. 6: Comparison of actual and predicted transmitted waves at room temperature

of hexagonal Si_3N_4 and an amorphous phase of thin grain-boundary layer or at grain-boundary triple junction. From macroscopic scale, any viscoelastic phenomenon may be represented by a combination of many Maxwell type bodies. A two-parallel Maxwell-body model of Fig.9 is suggested to describe the internal friction of Fig.7, and the springs and dashpots is related to the grain and amorphous phase in the microstructure. Because the grain is much more stable than the amorphous phase at elevated temperature [6], we assume that only one softening effect of amorphous phase come to effect in the test temperature range, the internal friction of peak and background components represent the different stages of the softening of the amorphous phase in the microstructure. On the basis of the assumption, the viscosity of the dashpots in Fig.7 is controlled by the same the thermal activation mechanism is of Arrhenius form,

$$\eta_1 = \bar{\eta}_1 \exp\left(\frac{Q}{RT}\right) \quad \eta_2 = \bar{\eta}_2 \exp\left(\frac{Q}{RT}\right) \quad (8)$$

where Q is the thermal activation energy, R is the gas constant and T is the absolute temperature, $\bar{\eta}_1$, $\bar{\eta}_2$ are constants. Hence, the complex modulus, M^* , and internal friction, δ , of the two-parallel Maxwell-body model of Fig.9 is

$$M^* = M_1 + iM_2 = \frac{E_1}{1 - \frac{iE_1}{\eta_1\omega}} + \frac{E_2}{1 - \frac{iE_2}{\eta_2\omega}}$$

$$\delta = \frac{M_2}{M_1}$$

$$E = E_1 + E_2 \quad (\text{at room temperature}) \quad (9)$$

Fig. 7: Curve of internal friction as temperature rises

Fig. 8: Change of wave velocity as temperature rises

Fig. 9: Two-parallel Maxwell-body model

Table 1: Test results and model parameters

wave velocity c (m/s)	9500
elastic modulus E (GPa)	288
frequency f (kHz)	5.93
Activate Energy Q (kcal/mol)	50
E_1 / E_2 ($E = E_1 + E_2$)	0.00761
$\bar{\eta}_2 / \bar{\eta}_1$	5.00×10^5
$2\pi\bar{\eta}_1 / E_2$	1.96×10^{-9}

The thermal activation energy as well as the parameters in the two-parallel Maxwell-body model in Fig.9 can be evaluated by a curve fitting based on Eqn.(8) and (9). Some deduction from Eqn.(9) gives

$$\frac{E_1}{E_2} = 2\delta^* \left[\delta^* + \sqrt{1 + (\delta^*)^2} \right] \quad (10)$$

where δ^* is the maximum value of the internal friction at the peak. Moreover, the peak part and background part in Fig.7 are mainly determined by the value of $\bar{\eta}_1/E_2$ and $\bar{\eta}_2/E_2$, respectively.

All the model parameters determined from the curve fitting is listed in Tab.1. The comparison of internal friction predicted by Eqn.(9) and test data is shown in Fig.7.

4. CONCLUSION AND DISCUSSION

A new test method for determining viscoelastic mechanical property by using SHPB tests of a long length specimen is proposed. It is useful for elevated temperature tests as well as viscoelastic material of low internal friction.

Elevated temperature SHPB tests of dense Si_3N_4 ceramics is performed. The internal friction at the frequency of 5.9 kHz is evaluated. It is found that the internal friction under axial loading also shows a feature of peak and background components as that under shear loading.

Assuming that a single thermal activation mechanism controls deformation of dense Si_3N_4 ceramics at elevated temperature, i.e., only softening of amorphous phase is taken into consideration, a two-parallel Maxwell-body model is proposed to describe the viscoelastic behaviors of dense Si_3N_4 ceramics under axial loading. The thermal activation energy of dense Si_3N_4 ceramics can be evaluated according to the curve fitting process.

As the temperature distribution along the specimen is not considered, as well as the incident stress wave is not a perfect single frequency harmonic stress wave, the test results should be taken on a qualitative basis.

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