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Mechanical spectroscopy of creep appearance in fine-grained yttria-stabilized zirconia

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Abstract

Mechanical spectroscopy or internal friction measurements were performed at high temperature in 3 mol% yttria-stabilized tetragonal zirconia. The mechanical loss spectrum is composed of a relaxation peak and a high temperature exponential background. It is shown that the transition from the peak to the exponential background accounts for creep appearance in the material. Mechanical loss is interpreted as due to grain boundary sliding. The associated activation enthalpy was found to be of 540 kJ/mol. In the low stress creep regime involved here a stress exponent of 2 and a grain size parameter of 1 are reported. A threshold stress behavior was observed, which can be associated with the obstacles limiting grain boundary sliding in this low stress regime.

Keywords: Creep; Grain boundaries; Mechanical spectroscopy; Sintering; ZrO_2

1. Introduction

High temperature plastic or even superplastic deformation in fine-grained ceramics is generally interpreted by grain boundary (GB) sliding.[1] This sliding process is often lubricated by an amorphous intergranular phase, which results from the sintering aids. However extensive GB sliding needs "accommodation processes" such as diffusion or dislocation motion in order to prevent any cavitation.[2,3] The deformation mechanisms have been described by different theoretical models leading to different values of the parameters, which enter the standard equation for high temperature strain rate $\dot{\varepsilon}$:

$$\dot{\varepsilon}(\sigma, T) = A\cdot\frac{Gb}{RT}\cdot\left(\frac{\sigma}{G}\right)^{n}\cdot\left(\frac{b}{\mathrm{d}}\right)^{p}\cdot D_0\cdot\exp\left(-\frac{Q}{RT}\right) \qquad (1)$$

where G is the shear modulus, b the Burgers vector, D_0 the limit diffusion coefficient, n the stress exponent, σ the applied stress, p the grain size parameter, d the grain size, Q the apparent activation enthalpy, R the gas constant and A a material constant.

As it concerns yttria-stabilized tetragonal zirconia (Y-TZP), recent papers[4,5] reported that the interpretation of creep in the low stress regime is still unclear. On the one hand, low stress creep could be interpreted as GB sliding after activation of a threshold stress.[4] On the other hand, authors have interpreted the low stress deformation of Y-TZP as Coble GB diffusion.[5]

As GB sliding dissipates energy, the measurements of mechanical loss[6] are well suited to study such a process. It has been observed that the relative displacement of grains separated by an intergranular glassy film gives rise to a mechanical loss peak, which evolves into an exponential background at higher temperature.[7] When the amount of the intergranular glassy phase is higher, the damping level is globally higher and so is the creep rate. When part of the amorphous phase forms amorphous pockets, a mechanical loss peak related to dissipation in these pockets has been observed.[8] Such a peak may account for a toughness improvement by energy dissipation in the glassy pockets, the surrounding skeleton being responsible for creep resistance.

Results of mechanical spectroscopy obtained at high temperature in 3 mol% yttria stabilized zirconia (Y-TZP) are shown in the present paper and analyzed in comparison with the creep behavior of these ceramics. It will be shown that mechanical spectroscopy is an alternative way to measure the parameters of Eq. (1).

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2. Mechanical spectroscopy[6]

Mechanical spectroscopy is a measurement technique, which refers to the anelastic behavior of materials. Elasticity is associated with the Hooke's law: $\sigma = E_u \cdot \varepsilon$ where E_u is the unrelaxed Young's modulus, σ the applied stress and ε the strain. Anelasticity differs from elasticity in that sense that the equilibrium strain is not instantaneously reached under the application of a stress. When stress is applied, the anelastic strain increases with time from zero to an equilibrium value. At equilibrium, a modified Hooke's law can be written: $\sigma = E_r \cdot \varepsilon$, where E_r is the relaxed Young's modulus. The evolution from one equilibrium state to a new one is called anelastic relaxation, and may be defined by two parameters, the relaxation time τ , and the relaxation strength $\Delta = (E_u - E_r) / E_u$.

From a microscopic point-of-view, the anelastic relaxation can be interpreted as being due to the movements of structural defects (elastic dipoles, dislocations, interfaces) from one equilibrium position defined at $\sigma = 0$ to another one defined at σ. The relaxation strength, Δ, is then proportional to the concentration of defects, which are relaxing; the relaxation time τ accounting for their mobility.

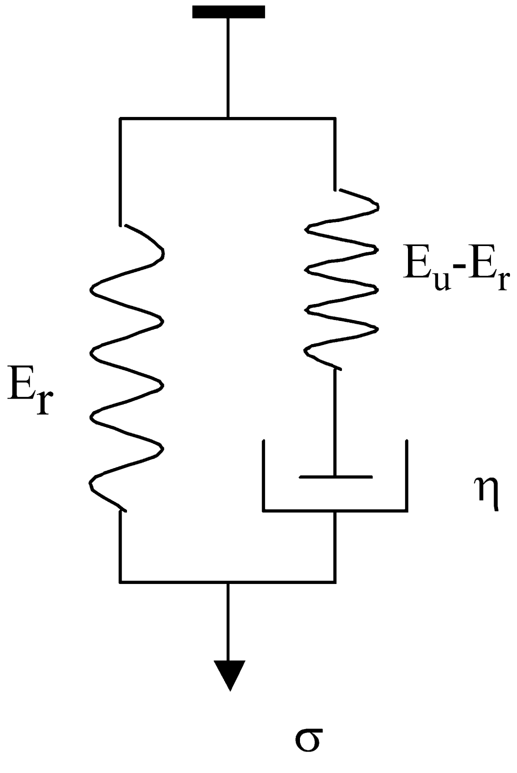

Fig. 1. Three-parameter rheological model, which accounts for the anelastic behavior of solid submitted to stress σ (spring constants are given in terms of E_u and E_r, the unrelaxed and relaxed elastic modulus, respectively, and η is the dashpot viscosity).

From a rheological point-of-view, the solid can be represented by the model in Fig. 1. The spring "E_r" in parallel with the dashpot of viscosity η is essential to the recoverable nature of the anelastic strain. If this spring does not operate, the anelastic strain does not reach an equilibrium value and creep strain is continuously accumulated. The model in Fig. 1 is representative of the standard anelastic solid, the equation of which is:[6]

$$\sigma + \tau \cdot \dot{\sigma} = E_r \cdot \varepsilon + \tau \cdot E_u \cdot \dot{\varepsilon} \tag{2}$$

If an alternating stress, of circular frequency ω, $\sigma = \sigma_0 \exp(i\omega t)$ is applied to the system, the linearity of the stress-strain relationships of anelasticity assures us that strain ε is periodic with the same frequency: $\varepsilon = \varepsilon_0 \exp[i(\omega t - \phi)]$, where ϕ is the phase lag of strain behind stress (due to anelasticity). Introducing these expressions of σ and ε in Eq. (2) leads to the following relationship:

$$\begin{aligned}\sigma &= \left[\left(E_u - \frac{E_u - E_r}{1+\omega^2\tau^2}\right) + i \cdot \left(\frac{(E_u - E_r) \cdot \omega\tau}{1+\omega^2\tau^2}\right)\right] \cdot \varepsilon \\ &= [E_1(\omega) + iE_2(\omega)] \cdot \varepsilon = E^*(\omega) \cdot \varepsilon \end{aligned} \tag{3}$$

where E_1 and E_2 are, respectively, the real and the imaginary part of the complex modulus E^*.

In metals and ceramics $\delta E < < E_u$, and the mechanical loss angle ϕ is given by:

$$\tan\phi = \frac{E_2}{E_1} = \frac{E_u - E_r}{E_u} \cdot \frac{\omega\tau}{1+\omega^2\tau^2} = \Delta \cdot \frac{\omega\tau}{1+\omega^2\tau^2} \tag{4}$$

Mechanical loss, tan ϕ, presents a maximum as a function of $\omega\tau$, centered at $\omega\tau = 1$. This maximum or peak gives us the relaxation strength, Δ, (height of the peak) and the relaxation time, τ, (position of the peak on the $\omega\tau$ axis). If several relaxation mechanisms are activated, the material exhibits a *mechanical loss spectrum* composed of damping peaks, which inform about microstructure dynamics. As most of the relaxation mechanisms are thermally activated,

$$\tau = \tau_0 \cdot \exp(Q/kT) \tag{5}$$

Q being the activation enthalpy. As a consequence, tan ϕ can be also measured as a function of temperature holding ω constant and the curve $\tan\phi = \tan\phi\,(T)$ is the mechanical loss spectrum described as a function of temperature.

The internal friction IF (or internal damping) of a material is defined as:

$$\mathrm{IF} = \frac{1}{2\pi} \cdot \frac{W_{\mathrm{diss}}}{W_{\mathrm{el}}} \tag{6}$$

where W_{diss} is the energy dissipated in a volume unit during one cycle of vibration and W_{el} is the maximum stored elastic energy per unit volume.

$$W_{\text{diss}} = \oint \sigma \cdot d\varepsilon = \pi \cdot \sigma_0 \cdot \varepsilon_0 \cdot \sin\phi = \pi \cdot E_2 . \varepsilon_0^2 \quad (7)$$

$$W_{\text{el}} = \int_0^{\varepsilon_0} \sigma \cdot d\varepsilon = \frac{1}{2} E_1 \cdot \varepsilon_0^2 \quad (8)$$

It follows immediately that:

$$\text{IF} = \tan\phi \quad (9)$$

3. Experimental data

Yttria-stabilized zirconia ceramics (3 mol%) were prepared from high purity powders (Tosoh, 3Y-TZP) by cold pressing (50 MPa) followed by air sintering at 1643 K for 3 h. Dense materials, with a final density higher than 94% of the theoretical value, were obtained by this method. In the as-sintered materials the mean grain size was about 0.29 µm.

The mechanical spectroscopy measurements were carried out in a differential, inverted torsion pendulum, working with forced vibrations.[9] In this apparatus, a plate of 3Y-TZP with dimensions $30 \times 4 \times 1$ mm^3 was mounted in series with a WC-Co specimen, which is used as an elastic reference. When torsion excitations are applied to the pendulum axis, the angular displacements of the sample and of the reference are converted into electrical signals through an optical system of lasers, mirrors and linear cells. Both signals are processed by a signal analyzer and from the phase difference and the ratio of their amplitudes, one can calculate the mechanical loss angle $\tan\phi$ and the normalized elastic shear modulus $G/G_{\text{reference}}$. Such measurements were performed under vacuum (10^{-4} Pa) as a function of temperature ($300 \div 1600$ K) at fixed frequencies, or as a function of frequency ($10^{-3} \div 10$ Hz) at fixed temperatures, or as a function of applied torsional stress ($0.1 \div 5$ MPa) at fixed temperatures and frequencies.

4. Results and analysis

Fig. 2 shows the results of mechanical loss measurements performed in 3Y-TZP at three different frequencies as a function of temperature. From 1350 K, the mechanical loss exhibits an exponential increase as a function of temperature. This phenomenon is thermally activated, because the curves are shifted towards higher temperature when the frequency is higher. In this temperature range, the mechanical loss does not exhibit a maximum (an internal friction peak) such as the peak corresponding with Eq. (4). The exponential form of the curves means that the anelastic strain has no limit in this temperature range. The observed behavior would correspond to a modified model in Fig. 1, where the E_r spring is absent. An exponential increase in the mechanical loss can be interpreted by the onset of creep in the material. Effectively, starting from Eq. (1), it is possible to calculate the mechanical loss due to creep by using Eq. (7).

$$W_{\text{diss}} = \oint \sigma \cdot d\varepsilon = \oint \sigma \cdot \dot{\varepsilon} \cdot \mathrm{d}t = A \cdot \frac{b}{\mathrm{RTG}^{n-1}} \cdot \left(\frac{b}{\mathrm{d}}\right)^p \cdot D_0 \cdot \exp\left(-\frac{Q}{RT}\right) \cdot \oint \sigma^{n+1} \cdot \mathrm{d}t \quad (10)$$

and by using Eqs. (6)–(9):

$$\tan\phi \cong A' \frac{G \cdot \sigma^{n-1}}{\omega \cdot d^p} \exp\left(-\frac{Q}{RT}\right) \quad (11)$$

with A' a material constant.

Eq. (11) shows that, if strain results from creep, the corresponding mechanical loss increases exponentially with temperature as observed in Fig. 2. Plotting mechanical loss in a logarithmic scale as a function of the inverse of the temperature yields a straight line (Fig. 3), the slope of which gives the activation enthalpy Q. For instance in Fig. 3, the slope of the 1 Hz curve yields a value of $Q = 166.5$ kJ/mol. This value is much smaller than the ones of about 500 kJ/mol reported in the literature[4,5] as it concerns creep of Y-TZP in the low stress regime. This indicates that the above analysis is simplistic and leads to apparent quantitative values.

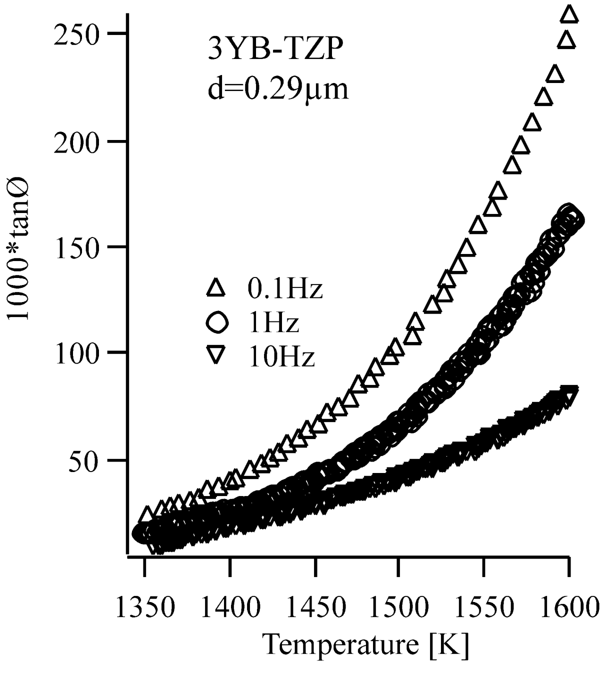

Fig. 2. Mechanical loss tanΦ measured as a function of temperature in 3 mol% yttria stabilized zirconia (3Y-TZP) at different frequencies. 0.1, 1 and 10 Hz.

Effectively, in real materials a distribution in the relaxation time τ has to be considered. Schoeck[10] showed that for constant relaxation strength the high temperature background can be expressed as:

$$\tan\Phi = \frac{C}{\omega^{\alpha}}\cdot\exp\left(-\frac{\alpha\cdot Q}{RT}\right) \tag{12}$$

where C is a constant and $1/\alpha$ is called the broadening factor. The α parameter can be obtained from the slope of log ($\tan\phi$) vs. $\log\omega$ plots. Fig. 4 shows isothermal spectra, in which the mechanical loss has been measured as a function of frequency from 1 Hz to 1 mHZ at different temperatures. For instance, in the case of the 1550 K spectrum, the value of α is found to be 0.315, which means a broadening factor of about 3.2. As the slope of the straight lines in Fig. 3 yields an apparent value of the activation enthalpy of $(\alpha.Q\ ) = 166.5$ kJ/mol, the correct value of Q would be of 530 kJ/mol, which corresponds better to the values obtained by creep tests in the low stress regime. Moreover it is well known in mechanical spectroscopy[6] that correct values of the activation enthalpy are obtained by reporting in an Arrhenius diagram the shift in temperature of the mechanical loss peak as a function of frequency. However, in the present case the spectra do not exhibit any peak, which would be well resolved with respect to the background. Looking at Fig. 4, one observes a shift towards higher frequency of the complete spectrum, when the temperature of the isothermal measurement is increased. One can consider the shifts in frequency $\Delta(\log(f))$ along the x-axis of the spectra in Fig. 4, which are necessary to superimpose two spectra taken at two different temperatures T_1 and T_2. Reporting these frequency shifts as a function of the difference $\Delta(1/T) = 1/T_1 - 1/T_2$ yields an Arrhenius plot (Fig. 5). The activation enthalpy measured by this "temperature-frequency" shifts of the whole mechanical loss spectrum yields a value of 540 kJ/mol.

Moreover, superposing all the shifted spectra of Fig. 4, one obtains a sort of "master curve" (Fig. 6), that means a generic spectrum which, together with the associated activation energy, contains the same information as all the spectra in Fig. 4 together. The master curve in Fig. 6 shows that the high temperature spectrum of Y-TZP is composed of a relaxation peak, which turns into an exponential background at low frequency when creep mechanisms can take place.

The effects of the oscillation amplitude have also been tested. Fig. 7 shows two 1550 K isothermal spectra measured at two different stress amplitudes. At low amplitude, one observes that the mechanical loss spectrum is effectively composed of a peak, located at about 0.1 Hz, and of an exponential background at lower frequency. The peak is not affected by the increase of the stress amplitude from 3.3 to 6 MPa. On the contrary the low frequency background depends on the vibration amplitude. The mechanical loss measured at low frequency (5 mHz) has been reported as a function of the applied stress in Fig. 8(a). Mechanical loss shows a weak dependence on the vibration amplitude for stress

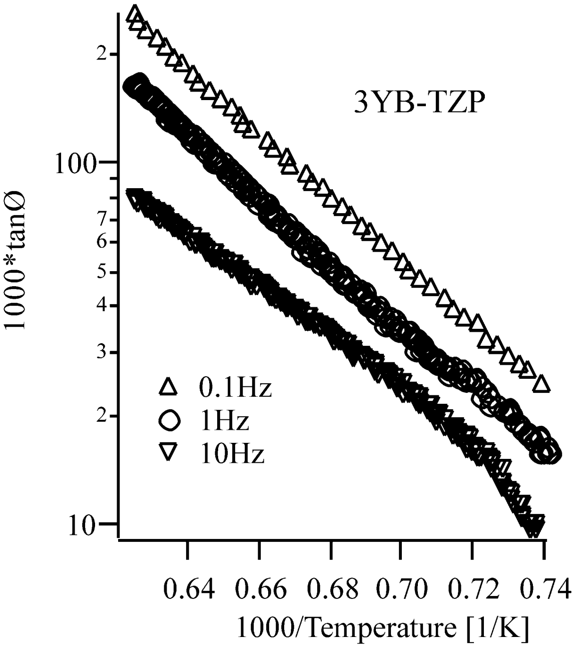

Fig. 3. High temperature mechanical loss curves of Fig. 2 plotted in a logarithmic scale as a function of the inverse temperature. The straight lines show that tanΦ has an exponential form. The slope of the lines yields an apparent value of the activation enthalpy of about 170 kJ/mol.

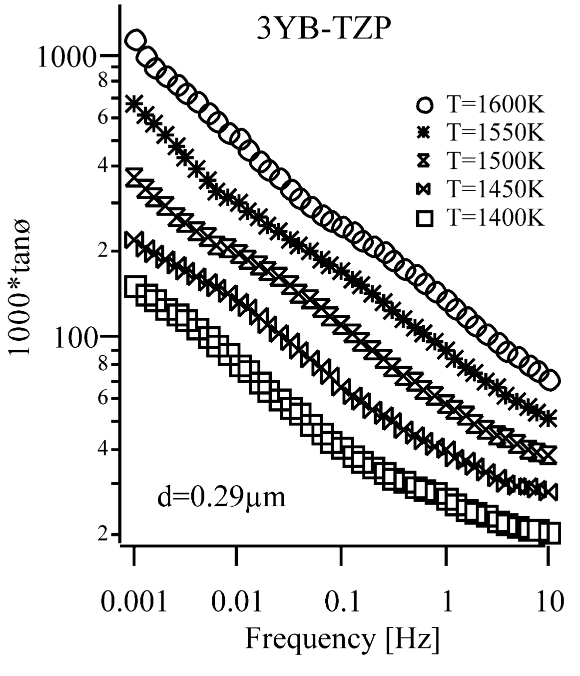

Fig. 4. Isothermal mechanical loss spectra of 3Y-TZP performed at different temperatures such as indicated on the graph, i.e. between 1127 and 1327 °C.

lower than about 1.5 MPa. For higher stresses a linear increase of damping with stress is observed. In this low stress regime, the transition from stress independent and stress dependent damping allows one to define a threshold stress. This threshold behavior is still more evident in the result of Fig. 8(b) obtained by L. Donzel[11,12] in a 2Y-TZP specimen. Here the threshold stress would be about 4 MPa. In the stress dependent damping, the stress exponent measured in Fig. 8(a) (slope of the curve) is 0.9, that means about 1. By considering Eq. (12), one can deduce that *n* is 2. The grain size dependence was also measured and published elsewhere.[13] The obtained results showed that grain size exponent in this low stress regime was about 1.

5. Discussion

Anelastic relaxation is interpreted as the reversible movements of structural defects from one equilibrium position to another one after the application of a stress. As high temperature plastic deformation of polycrystalline ceramics is associated with GB sliding, it seems reasonable to link their mechanical loss spectrum with GBs. A theoretical model[13] was developed for interpreting the high temperature mechanical loss of Y-TZP as due to the relative sliding of grains of hexagonal shape separated by an intergranular viscous layer. Two forces play a role in this process, which correspond to the elements of the rheological model in Fig. 1. The dissipative force corresponding to the dashpot in Fig. 1 is due to the viscosity of the intergranular layer; the restoring force corresponding to the "E_r spring" in Fig. 1 is due to the elasticity of the adjacent grains, which limit the sliding at the triple point junctions. Such a model accounts for a relaxation

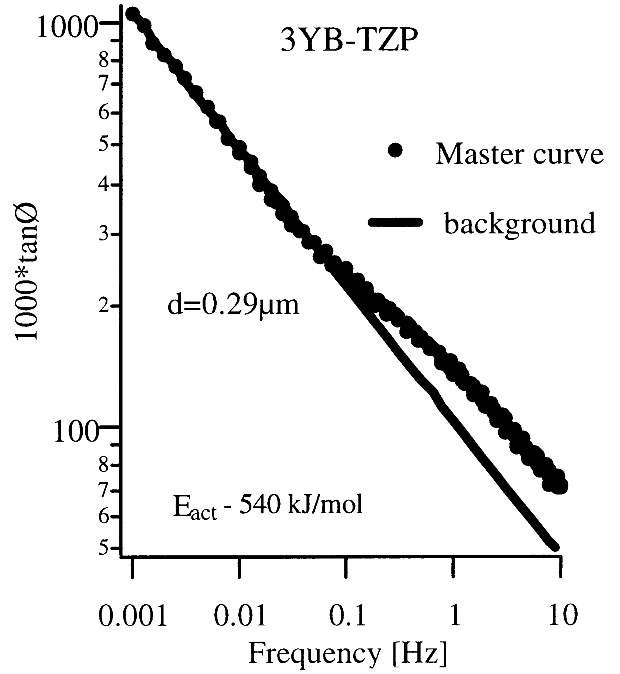

Fig. 6. Master curve built from the spectra of Fig. 4: the mechanical loss spectrum is composed of a peak and an exponential background at low frequency.

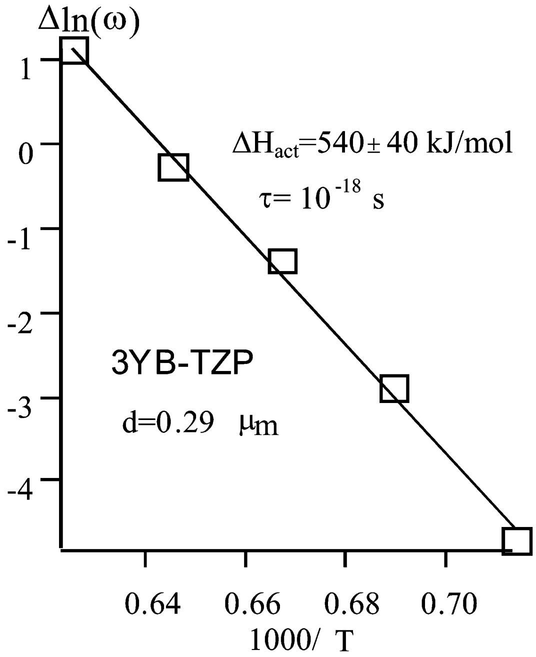

Fig. 5. Arrhenius plot obtained from the "temperature-frequency" shifts of the whole mechanical loss spectrum (Δln(ω) as a function of Δ(1/T)) yields an activation enthalpy of $Q = \Delta H_{act} = 540$ kJ/mol).

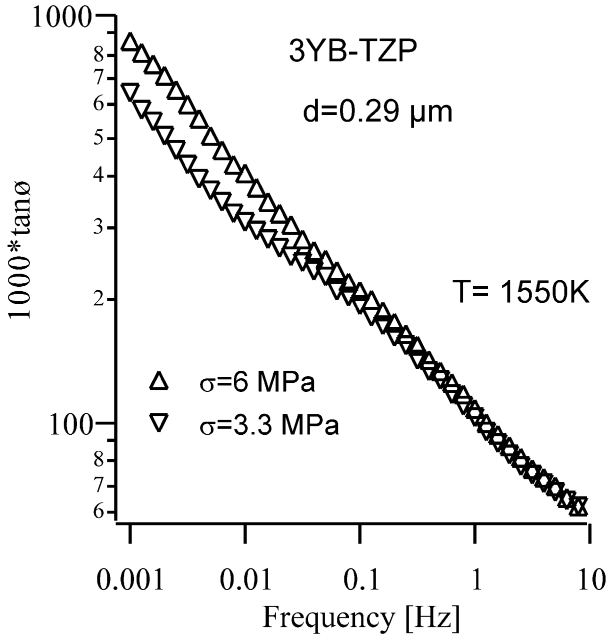

Fig. 7. Effect of the vibration amplitude on the mechanical loss spectrum at 1277 °C. Two curves have been reported for stress amplitudes of 3.3 and 6 MPa, respectively. Amplitude dependence is observed only in the low frequency range (exponential background).

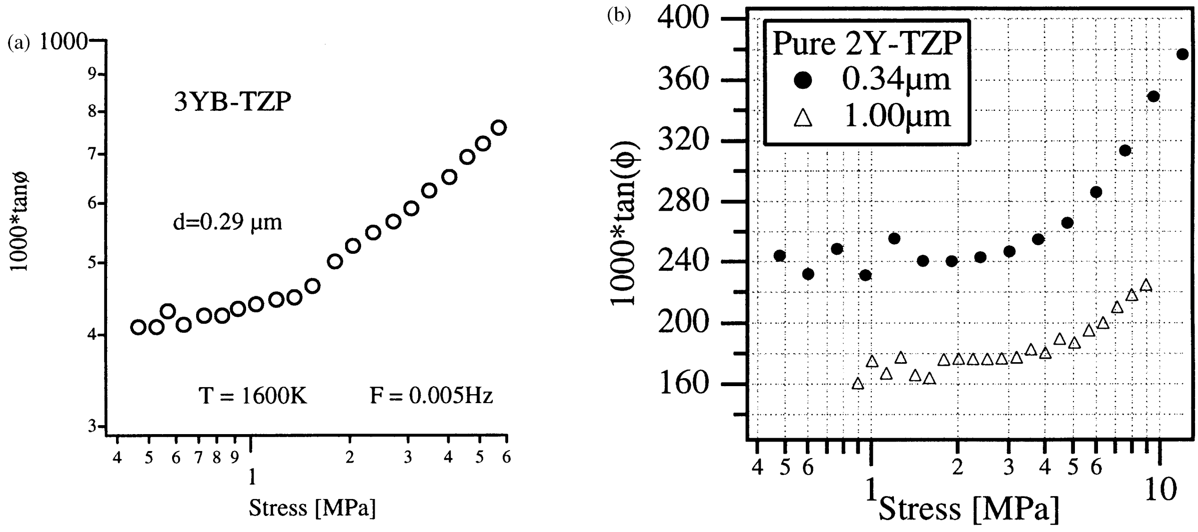

Fig. 8. Dependence of tanΦ on the stress amplitude at low frequency and high temperature. (a) In 3Y-TZP, at 5 mHz and 1327 °C, a threshold effect is observed at about 1.5 MPa. (b) In 2Y-TZP, at 1 mHz and 1267 °C, a threshold effect is observed at about 4 MPa.

peak. However in the obtained spectra (Figs. 2 and 4) one does not observe a well-resolved peak. The master curve in Fig. 6, which is a summary of all the measured spectra, shows that the observed peak is strongly bound to an exponential background at low frequency. It is possible to analyze the spectrum as composed of a relaxation peak, which evolves into an exponential background at low frequency. The precedent model can interpret such a behavior, by considering that the restoring force due to elastic interaction at the triple points decreases (softening of the E_r spring in Fig. 1) when temperature raises or when frequency decreases. The rheological model in Fig. 1 shows clearly that if the E_r spring does not operate, a creep behavior is observed. In other words, if the E_r spring constant goes to zero, there is no limitation to strain and as a consequence mechanical loss increases continuously with temperature or when frequency decreases. The signature of creep in the mechanical loss spectrum is an exponential form of damping (often called exponential background) as a function of temperature for measurements at fixed frequency or as a function of frequency for isothermal measurements.

A relaxation peak is representative of an anelastic behavior, in which a restoring force limits strain. In the case of GB sliding in ceramics, a well-resolved peak would be observed only if the pinning centers like GB asperities or triple point junctions are strong obstacles. For instance, a well-resolved peak was observed in silicon nitride due to the anelastic relaxation in the glassy pockets, the surrounding skeleton being responsible for the restoring force.[8]

In the case of Y-TZP zirconia, the regular shape of the grains does not provide strong pinning centers at the triple points. The mechanical loss spectrum can be interpreted as follow. The mechanical loss peak would account for the initial GB sliding limited by GB ledges or at the triple point junctions. GB sliding occurs at high temperature when the GB viscosity is low enough. However at sufficiently high temperature and low frequency, the probability to cross the above mentioned pinning centers increases and GB sliding is no more limited. It extends more and more when temperature raises or frequency is lowered. As a consequence, a transition from peak to exponential background is observed in the mechanical loss spectrum as an indication of the onset of creep.

In the peak domain, the mechanical loss does not exhibit any stress dependence (Fig. 7). This leads to a stress exponent of $n = 1$, similar to the one reported in the low stress regime by Jimenez-Melendo and Dominguez-Rodriguez.[4] In most of the cases, when the mechanical loss does not depend on the vibration amplitude, one has to do with a diffusion controlled atomic process. Recently, Charit and Chokshi[5] interpreted the low stress creep as controlled by a Coble GB diffusion process. However in that case the grain size parameter "p" was found to be about 3, a value predicted by the Coble creep model. In the present case, the dependence of the mechanical loss on the grain size leads to a value of $p \cong 1$.[13] This dependence of the mechanical loss on the grain size is in good agreement with the theoretical model of the anelastic relaxation due to GB sliding.[13] Moreover in the framework of the model, the mechanical loss peak does not depend on the stress amplitude, in good agreement with the obtained results (Fig. 4). On the contrary, Fig. 4 shows that the low frequency exponential background depends on stress amplitude. This is in accordance with Eq. (11), which yields the theoretical expression of tanϕ in the case of creep. The dependence of the exponential background on stress amplitude is only observed after a threshold stress of about 1.5 MPa (Fig. 5). In the domain of the amplitude dependence, a linear relation between tanΦ and the stress is observed. According to Eq. (11), this yields a value of $n=2$. This would argue for an interface reaction mechanism, which would control the extensive GB sliding.[5] In the peak domain (Figs. 6 and 7), the interface reaction would not be activated and then sliding is limited. For lower frequency than the peak, the period of oscillation is long enough to allow interface reaction and consequently a longer path for GB sliding: creep occurs. The restoring force, which appears in the relaxation model, would be overcome when the interface-reaction can be activated during one period of vibration, i.e. at sufficiently low frequency and high temperature.

The low frequency exponential background as it appears in isothermal measurements like in Fig. 7 allows one to study the mechanisms, which control creep in the low stress regime. In the present case, this exponential background shows stress dependence with a threshold stress. This threshold effect is similar to the one reported by Jimenez-Melendo and Dominguez-Rodriguez[4] for creep tests. Of course the level of the threshold stress of 1.5 MPa observed here in 3Y-TZP is lower than the one of 4.9 MPa deduced from creep.[4] On the other hand, this threshold stress of 4.9 MPa obtained in 3Y-TZP is of the same order of magnitude as the one observed in Fig. 8(b) for 2Y-TZP.[11,12]

Considering the earlier results, a reasonable interpretation of the mechanical loss spectrum would be based on GB sliding mechanism. A threshold stress is associated to this mechanism at the transition between local sliding (mechanical loss peak) and extensive sliding at the appearance of creep (exponential background).

6. Conclusions

At high temperature the mechanical loss spectrum of Y-TZP zirconia is composed of a relaxation peak, which evolves into an exponential background at higher temperature or lower frequency. The appearance of creep is associated with the transition "peak-exponential background". The exponential background shows a stress amplitude dependence with a threshold behavior. For stresses higher than the threshold stress, the stress exponent was found to be 2, the grain size parameter being 1 and the activation enthalpy 540 kJ/mol, respectively. The origin of damping is well accounted for by a theoretical model of GB sliding. GB sliding is limited in the peak domain by a restoring force. When this restoring force can be overcome, sliding is no more limited and mechanical loss increases exponentially. The threshold stress would be associated with the obstacles restricting the GB sliding.

Acknowledgements

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