Inelastic deformation of ceramics based on zirconium dioxide

E E Deryugin1,*, N A Narkevich1, I A Danilenko2 and Z Shmauder3

1Institute of Strength Physics and Materials Science of the SB RAS, Russia, Tomsk
2Donetsk Institute of Physics and Engineering of the NAS, Ukraine, Donetsk
3Institut für Materialprüfung, Werkstoffkunde und Festigkeitslehre, Universität Stuttgart, Stuttgart, Deutschland

*dee@ispms.tsc.ru

Abstract. The reasons for the nonlinear stage of deformation of two-cantilever specimens with a chevron notch under loading by the wedge method are investigated. Three main reasons are analyzed: plastic deformation, stable crack extension in the chevron notch zone, and phase transformation induced by stress concentration. It is shown that the main reason for the inelastic behavior of ceramics is transformation.

1. Introduction

Materials based on zirconium dioxide ZrO2, partially stabilized by yttrium oxide Y2O3, stand out among other structural ceramics with high strength values and increased crack resistance. The so-called transformation-hardening effect is characteristic for zirconium ceramics [1-4]. Mechanical stresses at the top of a growing microcrack initiate a phase transition from the tetragonal (t) modification to the monoclinic (m) modification. The loading diagrams of ceramics based on zirconium dioxide, loaded by the wedge method of two-cantilever specimens with a chevron notch, reveal a long stage of inelastic deformation [5], which is almost 40% of the contribution of elastic deformation. A characteristic feature of the loading curves is a significant relaxation of force P acting on the deflection of the cantilevers. The beginning of relaxation is characterized by the value of force Psr and determines the end of elastic and the beginning of inelastic deformation of the ceramics. The qualitative form of the curves is similar to the ‘tooth’ of yield on the loading curves of flat polycrystalline iron samples [6]. Presence of an inelastic stage of deformation indicates intense relaxation processes in the chevron notch zone, which may be caused by various reasons. This is, first of all, plastic deformation, stable crack extension, or phase transformation.

This article clarifies possible reasons for the inelastic behavior of ceramics based on zirconium dioxide stabilized with yttrium oxide.

2. Features of load diagrams

Ceramics obtained by sintering the powder for an hour at a temperature of 1600 °C was chosen as the research material. The test specimens had a notch length up to chevron \( l_0 = 24 \text{ mm} \), the section of each console \( ab = 4 \times 4 \text{ mm}^2 \), chevron angle \( \alpha = \pi/4 \). The notch thickness did not exceed 0.3 mm, \( a \) is sample thickness, \( b \) is console thickness (Figure 1). The tests were carried out by wedging two-cantilever ceramic specimens with a chevron notch with the composition ZrO2 + 3%Y2O3 и ZrO2 +
3%Y₂O₃ + 2wt%Al₂O₃. Experimental diagrams of ZrO₂+3%Y₂O₃ ‘pressure force on wedge F – displacement of wedge λ’ reveal a long stage of nonlinear behavior of the material before specimen destruction. Figure 1 shows diagrams for two of the six specimens with the largest difference in the nonlinear stage onset. Duration of the linear stage of deformation from sample to sample varies over a wide range. This is, apparently, due to the fact that when a mechanical incision is made, microcracks accidentally appear at the chevron. Another reason may be due to the difference in frictional forces between the wedge and the specimen. It is obvious that, with the same deflection force of specimen P cantilevers, the greater the friction coefficient, the greater pressure force F on the wedge.

Figure 1. Specimen with a chevron notch.

A specific feature of these diagrams is that the transition to the inelastic stage of deformation is clearly recorded in the form of a sharp decrease in the pressure force on the wedge. This effect is especially pronounced in ceramics ZrO₂+3%Y₂O₃+2wt%Al₂O₃. Figure 3 shows six diagrams of chevron notched specimens tested to failure. Experimental diagrams $F - \lambda^*$, as in the previous case, reveal a characteristic stage of nonlinear behavior of the material. However, in this case, the onset of inelastic deformation is associated with a sharp drop in force $F$. For definiteness, the start of relaxation of the pressure force on the wedge was designated as $F = F_{sr}$.

Figure 2. Diagrams of loading for ceramics ZrO₂+3%Y₂O₃.

Figure 3. Diagrams of loading for ceramic wedge ZrO₂+3%Y₂O₃+2wt%Al₂O₃.
Loading of the specimens by the wedge method was carried out according to the method described in [7]. Transition from the pressure force on wedge $F$ to force $P$ bending a separate cantilever of a two-cantilever specimen was calculated according to the formula

$$P = \frac{F \cdot \cos \gamma}{2(\sin(\beta/2) + \mu \cdot \cos(\beta/2))},$$  \hspace{1cm} (1)$$

where $\mu$ and $\gamma$ – respectively, the friction coefficient and the angle between the wedge and the console. The $\mu$ value was determined from the experiment using the equation

$$\mu = \frac{\Delta F}{\Delta \lambda^*} \cdot \frac{1}{E \cdot \sin(\beta/2)} \left( \frac{a}{b} \right)^2 \frac{1}{2} \left( 1 + \frac{a \cdot \cotg \frac{\alpha}{2}}{2} \right)^{-1} \frac{b}{2},$$  \hspace{1cm} (2)$$

where $E$ – Young’s modulus, $\Delta F/\Delta \lambda^*$ – the slope of the elastic loading line in the experimental diagram ‘pressure force on wedge $F$ – displacement of wedge $\lambda^*$’, $\beta = 9°$ – wedge opening angle. The relationship between $\lambda^*$ and the opening of cantilevers $\lambda$ obeys the equation $\lambda = \lambda^* \cdot \cotg(\beta/2)$.

Table 1 shows values of force $F_{sr}$, rigidity of the wedge-sample system $\chi = F/\lambda^*$ and values of friction coefficients $\mu$, calculated according to expression (2).

| Specimen No. | 1   | 2   | 3   | 4   | 5   | 6   |
|--------------|-----|-----|-----|-----|-----|-----|
| $F_{sr}$, N  | 27.987 | 20.584 | 24.079 | 23.748 | 23.285 | 20.54 |
| $\chi$, N/mm | 46.968 | 36.83 | 39.32 | 44.748 | 40.018 | 39.50 |
| $\mu$        | 0.158 | 0.103 | 0.114 | 0.145 | 0.125 | 0.154 |

Figure 4 shows the loading diagrams ‘cantilever deflection force $P$ – notch opening $\lambda^*$’, obtained using equations (1) and (2). The curves are numbered here in decreasing order of the start of relaxation of the force $P_{sr}$.

Comparison with Figure 3 shows that the scatter of quantitative characteristics of the curves has significantly decreased. Nevertheless, the difference in the upper limit of elasticity from sample to sample, regardless of the friction coefficient, remained significant.

A characteristic feature of the loading curves is a significant relaxation of force $P$ acting on the deflection of the cantilevers. The onset of relaxation is characterized by the value of force $P_{sr}$ and determines the end of elastic and the onset of inelastic ceramic deformation. The qualitative form of the curves is similar to the ‘tooth’ of yield on the loading curves of flat specimens of polycrystalline iron [6].

The curves with high $P_{sr}$ values (curves 1 - 3) at the stage of inelastic deformation have a tendency to material softening. The deflection force at this stage changes either insignificantly (curve 2) or decreases with specimen loading (curves 1 and 3). Curves with lower $P_{sr}$ values (curves 4 - 6) show clear signs of strain hardening. This property is especially clearly expressed in the specimen with the
lowest $P_r$ value (curve 6). Thus, the qualitative behavior of the curves for specimens at the stage of inelastic deformation depends on the value of relaxation start $P_{sr}$.

Figure 4 shows that the maximum value of the deflection force of the specimen $P_{max}$ cantilevers corresponds to the values of the relaxation start $P_{sr}$ for curves 1-4. For curves 5 and 6, the maximum deflection force is observed at the stage of inelastic deformation. There is also a correlation between $P_{sr}$ and the amount of force relaxation $\Delta P_r$. With an increase in the start of relaxation $P_{sr}$, the value of relaxation of the force $\Delta P_r$ also increases.

Table 2 shows the main characteristics of the $P$–$\lambda$ diagrams: $P_{max}$, $\Delta P_r$, $P_f$, etc. The diagrams are numbered in order of decreasing value of relaxation of force $\Delta P_r$. The $P_f$ value determines the strength limit of the specimen, which corresponds to the moment of ceramic fracture. The notch opening of sample $\lambda$ consists of elastic $\lambda_e$ and inelastic (relaxation) $\lambda_r$ parts: $\lambda = \lambda_e + \lambda_r$. The value $\lambda_r/\lambda_e$ determines the value of the inelastic part $\lambda_r$ to the elastic part $\lambda_e$ in the process of relaxation of force $P$ by the value $\Delta P_r$. The $\lambda_e/\lambda_e$ value is a similar ratio, where $\lambda_e$ corresponds to the inelastic opening at the time of sample failure. Table 2 also shows compliance values $\eta$ of the tested specimens. On average, the specimens are characterized by value $\eta = 1.56 \pm 0.04$ $\mu$m/N. The small scatter indicates a slight difference in their elastic and geometric characteristics.

| Diagram No. | 1  | 2  | 3   | 4     | 5     | 6     |
|-------------|----|----|-----|-------|-------|-------|
| $P_{max}$, N| 60.777 | 59.887 | 55.916 | 55.438 | 55.951 | 55.060 |
| $\Delta P_r$, N | 10.618 | 7.964 | 6.552 | 5.077 | 3.583 | 2.828 |
| $P_f$, N | 47.955 | 48.566 | 50.755 | 50.585 | 51.640 | 51.755 |
| $\lambda_r/\lambda_e$ % | 21.75 | 17.18 | 10.09 | 8.94 | 6.98 | 6.87 |
| $\lambda_e/\lambda_e$ % | 43.40 | 40.78 | 41.38 | 40.71 | 41.48 | 40.49 |
| $K_{IC}$, MPa$\cdot$m$^{1/2}$ | 5.540 | 5.345 | 5.092 | 4.930 | 4.569 | 4.564 |
| $\eta$, $\mu$m/N | 1.541 | 1.584 | 1.591 | 1.574 | 1.551 | 1.516 |

Dependences of breaking force $P_f$ and the relative fraction of inelastic deformation $\lambda_r/\lambda_e$ on the value $\Delta P_r$ are shown in Figure 5. It is seen that the fracture force of the specimen $P_f$ decreases with increasing $\Delta P_r$. The relationship between $\lambda_r/\lambda_e$ and $\Delta P_r$ can be interpreted as a linear law.

Further loading leads to an increase in the inelastic component of the cantilever displacement. By the beginning of the specimen fracture, the inelastic opening of the notch reaches the value $\lambda_r$. The relative fraction of inelastic deformation $\lambda_r$ by the time of fracture is practically the same for all specimens, regardless of the value $\Delta P_r$ (Figure 6). It averages $41\pm2\%$ of the contribution of elastic strain $\lambda_e$. The experiment showed that an increase in the relaxation $\Delta P_r$ leads to a noticeable increase in the maximum deflection force $P_{max}$.

Figure 5. Dependencies $\lambda_r/\lambda_e$ and $P_f$ from $\Delta P_r$.

Figure 6. Dependencies $\lambda_r/\lambda_e$ and $\lambda_f/\lambda_e$ from $\Delta P_r$.

It should be noted that such a pronounced stage of the nonlinear behavior of ceramics during testing for 3-point bending has not been noted in the literature [8, 9].
The stress intensity factor $K_{IC}$ (SIF) was calculated as a characteristic of the fracture toughness of the material under the assumption that the inelastic deformation of the ceramics is due to phase transformation, and not to the crack expansion mechanism. In [5], it is shown that in this case SIF is proportional to the bending force $P$. As $\Delta P$ decreases, a gradual decrease in $K_{IC}$ from 5.54 to 4.56 MPa $\cdot$ m$^{1/2}$ is observed (Table 2).

The data obtained can be used as input parameters for modeling the non-elastic behavior of ceramic materials.

3. Effect of crack on nonlinear behavior of material

3.1. Compliance of a double-cantilever specimen with a chevron notch

If the nonlinear behavior is associated with stable crack propagation in the chevron notch zone, then this will lead to a decrease in specimen compliance. The compliance of a double cantilever specimen can be defined as the ratio of the elastic opening of the notch $2\lambda_e$ to the deflection force of the cantilevers $P$: $\eta = 2\lambda_e / P$, where $\lambda_e$ is the deflection of an individual cantilever.

According to the methodology [7] developed at the Institute of Strength Physics and Materials Science of the SB RAS in the laboratory of physical mesomechanics and non-destructive testing methods for determining the crack resistance of a material, the compliance of a specimen with a chevron notch is

$$\eta = \frac{2\lambda_e}{P} = \frac{8 \cdot 1.22 \left( \frac{l_0 + \Delta l}{b} \right)^3}{k},$$

where $l_0$ – notch length to chevron, $\Delta l$ – crack length in the chevron notch zone,

$$k = \frac{2\Delta l}{l_0} \left[ 1 - \frac{2\Delta l}{l_0} \right] \left[ \frac{4 + \frac{a}{l_0} \cotg \frac{\alpha}{2} + \frac{2\Delta l}{l_0} \cotg \frac{\alpha}{2}}{\frac{2 + \frac{a}{l_0} \cotg \frac{\alpha}{2}}{l_0}} \right]^2.$$

In the case of a straight cut, $k = 1$ and the compliance is calculated by the formula

$$\eta = \frac{2\lambda_e}{P} = \frac{8 \cdot 1.22 \left( \frac{l_0 + \Delta l}{b} \right)^3}{P}.$$  (4)

Figure 7 shows dependences of the compliance of a specimen with a chevron (curve 1) and straight (curve 2) notches from the crack length. The calculations were carried out using dimensions corresponding to the dimensions of real specimens, namely: $a = b = 5 \text{ mm}$, $l_0 = 25 \text{ mm}$, $\alpha = 45^\circ$, $E = 210 \text{ GPa}$ [4, 10]. The height of the chevron is $h = 6 \text{ mm}$ (more precisely 6.036 mm). The value $\Delta l = h$ corresponds to the situation when the crack has passed the entire chevron zone, and the notch has become rectilinear. Compliance of the specimen at this moment corresponds to the compliance of the specimen of $h + l_0$ length with a straight notch and is equal $\eta = 2.223 \mu m/N$.

Calculations show that with crack expansion, an increase in sample compliance always occurs. According to the definition, in the diagram ‘$P - \lambda_e$’ the sample compliance $\eta = 2\lambda_e/P$ is defined as a double cotangent of the inclination angle of the elastic loading line to the $\lambda_e$ axis. An increase in specimen compliance with an increase in the crack length means that during repeated loading, the slope of the elastic loading line on the ‘$P - \lambda_e$’ diagram will be less than during the first loading.

In the case of a straight cut in the interval $\Delta l$ from 0 to $h$, the compliance increases almost 2 times (more precisely, 1.91 times), from 1.162 to 2.223 $\mu m/N$. In the case of a chevron notch, the increase in compliance is 40% from the initial value of 1.598 at $\Delta l = 0$ to 2.223 $\mu m/N$ at $\Delta l = h$. Such a significant change in compliance with crack expansion can be easily detected experimentally.
In this regard, the study of dependence of the compliance of a ceramic sample ZrO₂+3%Y₂O₃+2wt%Al₂O₃ in the deformation process to failure was carried out.

### 3.2. Diagrams ‘P – λ’ under secondary loading

To determine compliance at the stage of nonlinear deformation, the specimen was first loaded to a certain degree of inelastic deformation, then the external load was quickly removed. After holding the sample in an unloaded state for 2 minutes, it was loaded again until fracture. Compliance change was determined by comparing the slopes of the elastic stage of the two diagrams.

Figure 8 illustrates two diagrams each for three samples, which were deformed first to a certain degree of nonlinear deformation, then deformed again before fracture. The first sample was unloaded almost immediately after the beginning (start) of the relaxation of the deflection force. The second was unloaded after a rather long stage of inelastic deformation. For the third sample, the load was removed shortly after reaching the maximum force before failure.

The change in compliance at the first (η₁) and the repeated (η₂) loading is presented in Table 3. It also shows the stress intensity factor $K_{IC}$, the maximum load before failure $P_{max}$ and the start of relaxation $P_{sr}$.

### Table 3. Specimen characteristics

| No. | $\eta_1$, µm/N | $\eta_2$, µm/N | $\eta_1/\eta_2$, % | $K_{IC}$, MPa m$^{1/2}$ | $P_{sr}$, N | $P_{max}$, N |
|-----|----------------|----------------|-------------------|------------------------|-------------|-------------|
| 1   | 2.887          | 2.862          | 0.86              | 4.974                  | 38.568      | 40.672      |
| 2   | 2.638          | 2.640          | 0.07              | 5.290                  | 36.777      | 44.316      |
| 3   | 2.164          | 2.196          | 1.48              | 5.015                  | 33.719      | 45.150      |

It follows from the data presented that at the stage of inelastic deformation the change in the compliance of the specimens does not exceed the experimental error (± 1%).

![Figure 7](image1.png)

**Figure 7.** Dependences of specimen compliance on crack length for chevron (curve 1) and straight (curve 2) notches.

![Figure 8](image2.png)

**Figure 8.** Loading of ceramics to varying degrees of inelastic deformation with repeated loading to fracture.
Thus, experiments do not confirm a noticeable change in the compliance of the samples during loading up to the moment of fracture.

Analysis of the experimental results revealed an interesting feature of the material, namely, the ability to undergo additional strain hardening upon repeated loading. In case of repeated deformation, the maximum deflection force of the cantilevers $P_{\text{max}}$ always exceeds the value $P_{\text{sr}}$. In this case, the more accumulated inelastic deformation, the greater the effect of strain hardening.

The presence of a crack in the zone of the chevron notch is not detected and the photographic image of the specimen during loading. Figure 9 shows photographs of a chevron notch at the final stage of specimen deformation. Immediately before fracture (Figure 9a), no signs of cracking or destruction are observed. Destruction occurs by spalling of one of the specimen arms due to initiation and rapid expansion of a crack along the chevron notch (Figure 9b).

**Figure 9.** Chevron notch: a - 2 s before destruction, b - destruction.

4. $t\rightarrow m$ transformation

Another reason for the nonlinear behavior may be the ability to phase transformation of ceramics ZrO$_2$+3%Y$_2$O$_3$+2wt%Al$_2$O$_3$. It is known that zirconium dioxide undergoes a polymorphic $t\rightarrow m$ transformation upon cooling, in which the tetragonal phase ($t$) transforms into the monoclinic modification ($m$). In this regard, the question of the influence of the phase transformation on the form of loading diagrams, and, consequently, on the parameters of the crack resistance of ZrO$_2$ ceramics, requires clarification.

To test this assumption, an X-ray structural analysis was performed. The phase composition of the ceramic specimens was investigated after fracture by X-ray diffractometry on a DRON-4M device in $\text{CoK}_\alpha$ radiation. The lateral face of the sintered ZrO$_2$ specimen before mechanical tests and the fracture region in the plane of the chevron notch after mechanical tests were investigated.

Analysis of the obtained diffraction patterns (Figure 10) indeed revealed formation of an $m$-phase with a monoclinic lattice in the fracture. Under loading, a structural $t\rightarrow m$ transformation occurs in the fracture zone, at which, according to our estimate, the increase in the volume of each of the transformed crystallites is $\sim 4.4\%$.

**Figure 10.** X-ray of ceramics ZrO$_2$+3%Y$_2$O$_3$. 

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Apparently, this mechanism plays a major role in the nonlinear behavior of ceramics ZrO$_2$+3%Y$_2$O$_3$+2wt%Al$_2$O$_3$. Indeed, the analysis of photographic images using a high-resolution Pentax K-5 camera showed that at the entire stage of the nonlinear behavior of the material, there are no signs of material destruction. The load drop to zero occurs as a result of crack initiation at the final stage of deformation.

5. Conclusion

Tests of double-cantilever specimens with a chevron notch by the wedge method showed that the inelastic deformation of ceramics based on zirconia stabilized by the addition of 3% yttrium oxide amounts to almost 40% of the total deformation of the material. A specific feature of the inelastic stage of deformation is the presence of a pronounced elastic limit, similar to a ‘tooth’ and a yield area in the tensile diagrams of flat polycrystals of low-carbon steel. There is a linear correlation between the relaxation value of the deflection force $\Delta P$ and the fracture force $P_f$ of the specimen.

It has been experimentally established that at the stage of inelastic deformation, there is no significant change in the compliance of the specimen. This eliminates the connection between inelastic deformation and the crack expansion mechanism. X-ray diffraction studies of the fracture surface revealed formation of an $m$-phase with a monoclinic lattice in the fracture, associated with the ability of ceramics ZrO$_2$+3%Y$_2$O$_3$ to phase $t \rightarrow m$ transformation. This is evidence in favor of the mechanism of phase transformation induced by stresses in the bulk of the material.

There is no information on the motion of dislocations in the structure of ZrO$_2$+3%Y$_2$O$_3$ at room temperature [11].

Thus, experiments convincingly show that the nonlinear stage in the loading diagrams of a ceramic specimen is associated not with dislocation plasticity or stable crack expansion, but with the development of a structural-phase transformation in the chevron notch zone.

The data obtained can be used as input parameters for modeling the non-elastic behavior of ceramic materials.

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