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### Publication Date

1988-06-01

Center for Advanced Materials

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## Effect of Temperature on the Densification/Creep Viscosity During Sintering

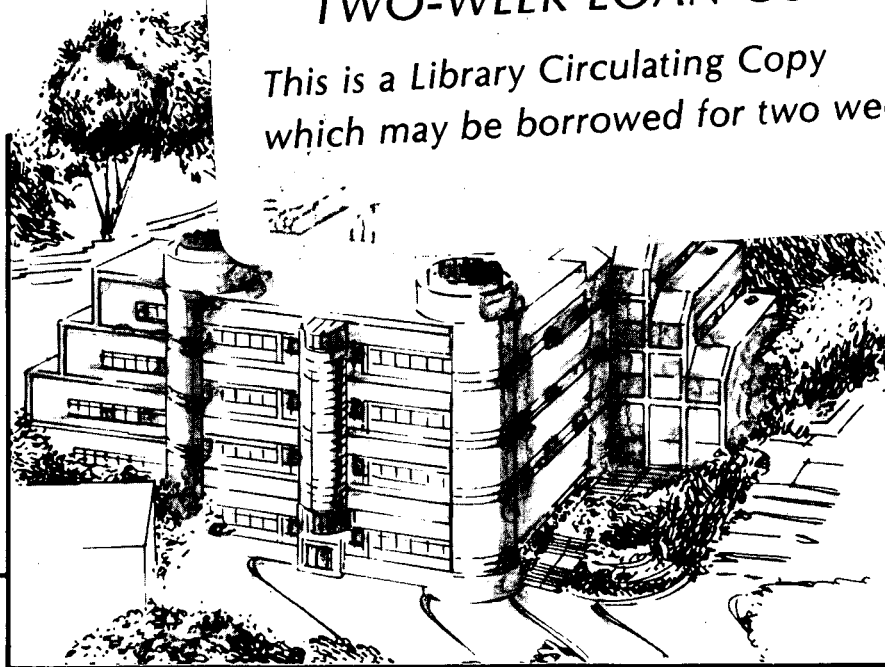
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EFFECT OF TEMPERATURE ON THE DENSIFICATION/CREEP VISCOSITY DURING SINTERING

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ABSTRACT

The densification and creep rates of polycrystalline zinc oxide and of alumina powder compacts have been measured simultaneously in a loading dilatometer, under constant heating rate and constant temperature conditions. The data show that the ratio of the densification to the creep rate is remarkably constant within a wide temperature range for densification, from the earliest stages of densification to at least the onset of the final stage. As a consequence, the ratio of the densification to the creep viscosity is nearly independent of temperature or sintered density. The analysis leads to a simple method for determining the simultaneous densification over the creep rate ratio. The implications of the result for the processing of heterogeneous ceramics and composites are discussed.

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\* Member, the American Ceramic Society.

## 1. INTRODUCTION

It is widely recognized that the densification behavior of powder compacts depends significantly on their uniformity, and efforts to achieve a high degree of uniformity in the initial structure of powder compacts have been very fruitful. In many if not most practical powders, however, agglomerate formation cannot be completely avoided. Further, in a number of important cases, such as the particulate composites, inhomogeneities are an essential part of the system. The presence of heterogeneities in the powder compacts lead to local, non-uniform densification rates which, in turn, produce stresses in the surrounding matrix that oppose densification. Unless these stresses are relieved by shear deformation (i.e. creep) they can lead to a significant reduction in densification rates and may exacerbate the development of deleterious crack-like voids around inclusions.<sup>1-9</sup> In addition, clustering of the dispersed phase will lead to the formation of extended skeletal segments of the inert particles, arresting macroscopic densification, as was discussed by Lange.<sup>10</sup> These clustering effects, rather than the backstresses generated by local densification rate mismatch, are likely to dominate the densification behavior at high inclusion content in particulate composites. If techniques were employed that prevented the formation of percolation networks of the inert phase, then the backstresses resulting from local densification rate mismatch would remain as the most significant factor impeding free sintering of particulate composite powder compacts.

It has been shown by De Jonghe et al.<sup>5</sup> and by Scherer et al.<sup>11</sup> that the backstresses arising during the densification of particulate composite powders are simply viscous, i.e., proportional to the instantaneous densification rate of the matrix. This leads to a matrix densification rate that is determined by the volume fraction of the dispersed inert phase and by the ratio of the creep viscosity over the densification viscosity:

$$\dot{\epsilon}_m = \dot{\epsilon}_{m0} / (1 + 4(\eta_c / \eta_d) f / (1 - f)) \quad \text{Eqn. 1}$$

where the superscript dot represents differentiation with respect to time, and where

$\dot{\epsilon}_m$  = densification rate of the matrix for the composite

$\dot{\epsilon}_{m0}$  = matrix densification rate at  $f=0$

$f$  = volume fraction of the hard inclusion

$\eta_c$  = creep viscosity of matrix

$\eta_d$  = densification viscosity of the matrix defined by

$$\dot{\epsilon}_{m0} = -(\Sigma/\Phi) / \eta_d, \text{ where } (\Sigma/\Phi) \text{ is the sintering stress}^5$$

The magnitude of the ratio of the creep viscosity over the densification viscosity of the matrix is therefore an important parameter in the densification of particulate systems in which the inclusion phase is uniformly distributed, even though it is unlikely to vary by orders of magnitude, as first postulated (Hsueh<sup>3</sup>, Raj<sup>2</sup>).

Practically, densification cannot be carried out isothermally, and it is well known that significant densification usually occurs by the time the isothermal sintering condition has been reached. Alternatively, non-isothermal processing methods might be considered to affect the microstructural evolution of the powder compact. Thus, the dependence of the ratio of the densification rate over the creep rate on temperature needs to be examined for non-isothermal conditions. From the ratio of the densification rate over the creep rate, the ratio of the creep viscosity over the densification viscosity may be inferred, allowing an assessment of the possible effects of heating schedules on densification of particulate composites.

Rahaman and De Jonghe have used the technique of loading dilatometry<sup>12</sup> to measure simultaneously the densification and creep rates in a number of

ceramic powder systems.<sup>13-19</sup> Generally, for each system, the densification and creep rates were measured at a single, isothermal sintering temperature. A selection of the data is shown in Fig. 1, where the ratio of the linear densification rate to the creep rate is plotted as a function of relative density. Over a wide density range, covering at least the intermediate stage of sintering, the ratio of the densification over the creep rate is almost independent of density. The value of this ratio decreases with decreasing green density and is system specific.

In a study on the creep-sintering of soda-lime glass powder compacts<sup>18</sup> conducted at two closely separated temperatures (580 and 605°C), the data indicated that the ratio of the densification to the creep rate was nearly independent of temperature (Fig. 1). The objective of the present paper is to examine the dependence of the densification and creep rates on temperature from experiments conducted over a wide temperature range for a constant heating-rate schedule. Data were obtained for two different crystalline powders: ZnO and Al<sub>2</sub>O<sub>3</sub>. For comparison, data were also obtained on the isothermal creep-sintering of alumina.

## 2. EXPERIMENTAL PROCEDURE

### a. ZnO

Zinc oxide\* powder compacts (6mm in diameter by 6mm) having a green density of  $0.44 \pm 0.01$  of theoretical were made by uniaxial compaction in a tungsten carbide die at  $\approx 10$  MPa. The same powder was used by the present authors in earlier sintering studies.<sup>7,13</sup>

The compacts were sintered in air in a loading dilatometer<sup>12</sup>, under

\*Reagent grade, Mallinckrodt Inc., Paris, KY

uniaxial loads of 0 or 7 N, at a constant heating rate of 4°C/min. The experiment was stopped at 1000°C, where densification was nearly complete. The load of 7 N, corresponding to an initial stress on the green compact of 0.24 MPa, was applied when the temperature had reached 500°C when shrinkage just commenced. The mass and dimensions of the samples were measured before and after sintering and the final densities were verified using Archimedes' method. In a separate set of experiments, sintering under the load of 7 N was terminated at temperatures between 500 and 1000°C, and the dimensions of these compacts were measured using a micrometer.

#### b. Alumina

Procedures similar to those for ZnO were used for the alumina powder\*. The powders were compacted in a die to a green density of  $0.52 \pm 0.01$  of theoretical. They were then sintered in air at constant heating rate, up to a maximum temperature of 1400°C. The loads of 0 and 7 Ns were applied from 1000°C on, at which point densification could not yet be detected. In addition to constant heating rate experiments, samples were isothermally sintered at 1350°C in air, for 2 hours, under a load of 7 N. These samples were inserted rapidly into the hot zone of the dilatometer and reached the sintering temperature in about 5-7 min. After the initial temperature transient the load was applied.

### 3. RESULTS

#### a. ZnO

Figure 2 shows the results for the axial true strain,  $\epsilon_z$ , versus temperature for the ZnO samples sintered at a constant heating rate of 4°C/min under zero applied stress and under an initial stress of 0.24 MPa. Each curve is the

\*Alcoa Chemicals, Pittsburg, PA.



average of two runs under the same conditions and each is reproducible to  $\pm 1\%$ . The results for the axial true strain,  $\epsilon_z$ , versus the radial strain,  $\epsilon_r$ , determined by terminating the sintering runs at temperatures between 500 and 1000°C, are shown in Fig. 3; each data point was obtained using a different sample. The shrinkage of the sample sintered under zero stress is seen to be almost isotropic.

Within the limits of experimental error, the applied stress caused no change in the density; the final density of the sample sintered under the initial stress of 0.24 MPa was 0.97 compared with 0.96 for the sample sintered under zero stress. The deformation of the sample under the action of the applied stress was therefore constant volume creep.

#### b. Alumina

Fig. 4 shows the axial true strain,  $\epsilon_z$ , versus the radial true strain,  $\epsilon_r$ , for the alumina powder compacts. Line A corresponds to the constant heating rate experiments, while line B corresponds to the isothermal experiments, both at an applied load of 7 N. Line B does not go through the origin, since some shrinkage has already occurred in the heat-up period, before isothermal conditions were reached and the load was applied. Line C corresponds to the zero load sintering.

#### 4. ANALYSIS

In this section the densification strain rate,  $\dot{\epsilon}_d$ , and the creep rate,  $\dot{\epsilon}_c$ , are related to the axial strain/radial strain plot,  $\epsilon_z$  versus  $\epsilon_r$ . From the analysis, some simplified experimental procedures emerge.

For a body loaded uniaxially and deforming uniformly it is readily shown that the linear densification strain is

$$3\epsilon_d = \epsilon_z + 2\epsilon_r \quad \text{Eqn. 2}$$

while at the same time, for the total axial strain,  $\epsilon_z$ :

$$\epsilon_z = \epsilon_c + \epsilon_d \quad \text{Eqn. 3}$$

from which

$$\epsilon_c = 2(\epsilon_z - \epsilon_r)/3 \quad \text{Eqn. 4}$$

where the meaning of the various strains is indicated in Fig. 5.

If the creep rate at constant uniaxial stress is linearly proportional to the densification rate then one must expect that

$$\dot{\epsilon}_z = k\dot{\epsilon}_r \quad \text{Eqn. 5}$$

The relative densification and creep strains as shown in Fig. 6 can be obtained from dilatometer data in Figures 2 and 3, and density measurements. The procedure is particularly simple in the case of constant volume deformation, as was the case here.

The densification strain rate,  $\dot{\rho}/3\rho$ , and the creep strain rate,  $\dot{\epsilon}_c$ , can be obtained as a function of temperature by fitting smooth curves through the data of Fig. 6 and differentiating according to Eqs. (6) and (7):

$$\dot{\rho}/3\rho = \dot{\epsilon}_d = (4/\rho)d\rho/dT \quad \text{Eqn. 6}$$

$$\dot{\epsilon}_c = 4d\epsilon_c/dT \quad \text{Eqn. 7}$$

T is the temperature, and the factor 4 arises from the constant heating rate of 4°C per minute that was used in the experiments when time is expressed in minutes.

Eqns. 4 and 5 are also valid as strain rate equations so that

$$\dot{\epsilon}_d / \dot{\epsilon}_c = (k+2)/2(k-1) \quad \text{Eqn. 8}$$

At fixed density, i.e.  $\dot{\rho}=0$ , constant volume creep requires that

$$\epsilon_c = -2\epsilon_{rc} \quad \text{Eqn. 9}$$

where  $\epsilon_{rc}$  is the radial component of the creep strain. The locus of  $\epsilon_z$  at some fixed density in a plot of  $\epsilon_z$  versus  $\epsilon_r$  is then a straight line with a slope of -1:2, as shown in Fig. 7a. The data for constant load creep should then follow a straight line in the  $\epsilon_z/\epsilon_r$  plot and intersect a constant density line when the sintered density with applied uniaxial stress equals that of the sintered density at zero load. This intersection for ZnO is shown in Fig. 7a, and shows at once that creep indeed occurs at nearly constant volume. Similar results are obtained for Al<sub>2</sub>O<sub>3</sub>. Near constant volume creep has been observed for all ceramics studied so far by low-load loading dilatometry. It is then possible, with considerable confidence, to identify the  $\epsilon_r$  value corresponding to a measured shrinkage strain,  $\epsilon_z$ , for a particular density, by making a construction as shown in Fig. 7b, without the need of measuring  $\epsilon_r$  directly.

As seen from Eqn. 8, the ratio of  $\dot{\epsilon}_d/\dot{\epsilon}_c$  is very sensitive to variations in the values of k. In the actual experiment, the data are collected at constant load rather than at constant stress. Since the sample cross

sectional area,  $A$ , changes from the initial one,  $A_0$ , as a result of densification and creep, the applied stress,  $\sigma$ , differs from the initial stress,  $\sigma_0$ ,:

$$\sigma = \sigma_0 A_0 / A \quad \text{Eqn. 10}$$

An estimate can be made readily of how much change in  $k$  would be associated with the changing stress for the experimental conditions adopted here, or what the error would be if the constant load experiment is simply treated as if it were at constant stress. To allow for this estimate the total strains are needed, and for the sake of illustration a simple creep and densification relationship, which materials such as ZnO obey well for a large part of the densification, may be adopted:

$$-\epsilon_d = B_d (\Sigma/\Phi) \ln(t) \quad \text{Eqn. 11}$$

and

$$-\dot{\epsilon}_c = B_c \sigma / t = B_c \sigma_0 (A_0/A) / t \quad \text{Eqn. 12}$$

where  $B_d$  and  $B_c$  are kinetic constants. At the same time one has:

$$A_0/A = (R_0/R)^2 = \exp(-2\epsilon_r) \quad \text{Eqn. 13}$$

when  $R_0$  and  $R$  are the initial and the instantaneous radius of the sample cross section. To a first approximation the creep rate may be expressed as

$$-\dot{\epsilon}_c = \sigma_0 B_c (1 - 2\epsilon_r) / t \quad \text{Eqn. 14}$$

At low loads, the creep strain can be taken to be much smaller than the densification strain (typically  $\epsilon_c < 0.2 \epsilon_d$ ). Then, with  $\epsilon_r \approx \epsilon_d$ , integration yields

$$\epsilon_c(t, \sigma) = \epsilon_c(t, \sigma_0) (1 - \epsilon_d)$$

Eqn. 15

Eqn. 15 can be used to correct  $k$  to constant stress. The necessary information is shown in Figure 8 where line 1 is the constant load line, line 2 is the isotropic line and line 3 is the constant density line. The correction to each  $\epsilon_r$  and  $\epsilon_z$  datum point for ZnO is shown in Fig. 9.

The values of  $k$  that follow from such an analysis are 1.33 for ZnO, and 1.29 and 1.30 for  $Al_2O_3$  done under constant heating rate and isothermal conditions respectively. Using Eqn. 8 this would then yield values of the densification rate over the creep rate as 5.00 for ZnO, and 5.67 and 5.50 for  $Al_2O_3$  all at a stress of .24 MPa. More accurate data can be obtained from the creep rate (converted to constant stress rates by compensating for the cross-sectional area change) and densification rate measurements, rather than from the total strain determination. The ratios of  $\dot{\epsilon}_d/\dot{\epsilon}_c$  obtained from the rate measurements are 4.5 - 5.0 for ZnO and 5.7-6.5 for  $Al_2O_3$ . The data for ZnO is shown in Fig. 10. The two methods for obtaining  $\dot{\epsilon}_d/\dot{\epsilon}_c$  are in close agreement. This shows the total strain determination is useful for generating values of  $\dot{\epsilon}_d/\dot{\epsilon}_c$  with minimum experimentation.

## 5. DISCUSSION OF RESULTS

Fig. 10 shows the ratio of the densification rate over the creep rate for ZnO. This ratio is nearly constant as a function of temperature, for the ceramics examined here: ZnO and  $Al_2O_3$ . The results of the isothermal loading dilatometry in Fig. 1 demonstrate that the creep rate over the constant-stress densification rate is also insensitive to density changes for a particular system. In addition, as seen in the  $Al_2O_3$  experiments, densification rate over the creep rate is equal in this experimental case for

both the isothermal condition and the constant heating rate condition. Therefore, the densification over the creep rate may be obtained reliably from a non-isothermal experiment, extending the measurement range to the earliest stages of densification. Deviations of the proportionality between the constant stress creep rate and the simultaneous densification rate would be manifested by the  $\dot{\epsilon}_z$  vs.  $\dot{\epsilon}_r$  data not extrapolating through the origin. Such a derivation is not apparent in Figs. 4 and 9; thus  $\dot{\epsilon}_d/\dot{\epsilon}_c$  is nearly constant from the onset of sintering. The last stages of densification remain unexplored to date, since accurate measurements become difficult at high densities.

The near-constancy of the densification rate over the creep rate under widely varying conditions of temperature and density are perhaps somewhat unexpected, and indicate at the same time that relatively little benefit is to be expected from non-isothermal densification schemes for particulate composites. The benefits that may result from non-isothermal heating schedules would, in fact, be those that are obtained in the improved sintering behavior or microstructure of the inclusion-free matrix.

The ratio of the creep viscosity over the densification viscosity does not follow directly from the measured values of the densification rate over the creep rate. To obtain these values from loading dilatometry experiments, it will be necessary to measure precisely the small density increases that result from the hydrostatic component of the applied uniaxial stress. In principle, this should be possible, since the hydrostatic component of the applied stress,  $\sigma_a$ , can be added simply to the sintering stress,  $\Sigma/\Phi$ :

$$\dot{\epsilon}_d(\sigma_a) = \{ (\Sigma/\Phi) + \sigma_a/3 \} / \eta_d = \dot{\epsilon}_d(\sigma_a=0) + \sigma_a/3\eta_d \quad \text{Eqn. 16}$$

so that with

$$\dot{\epsilon}_c = \sigma_a / \eta_c$$

Eqn. 17

one obtains

$$\eta_c / \eta_d = 3 \{ \dot{\epsilon}_d(\sigma_a) - \dot{\epsilon}_d(\sigma_a=0) \}$$

Eqn. 18

The measurements presented here were not sufficiently accurate to determine whether this ratio is indeed constant throughout the densification process. In one liquid phase sintering system, accurate measurements showed that the ratio was indeed constant throughout the intermediate stage of densification, and approximately equal to 2. Theoretical consideration of Scherer also lead to the conclusion that the ratio of the creep viscosity over the densification viscosity should be nearly constant and equal to 2. Further, the constancy of the densification rate over the creep rate would require some unlikely compensation mechanisms to be at work if the viscosity ratio were not constant. All these consideration lead to the conclusion that both the sintering stress,  $\Sigma/\Phi$ , and the creep viscosity over the densification viscosity,  $\eta_c/\eta_d$ , are constant from the very early stages to late into the intermediate sintering stage.

## 6. CONCLUSIONS

The results of the present study show that, when constant volume creep deformation occurs during sintering, the ratio of the densification to the creep rate is almost independent of temperature.

Within the useful temperature range for densification, the ratio of the densification to the creep viscosity is nearly independent of temperature.

The present data have important implications for the sintering of heterogeneous ceramics and composites. The matrix densification rate of a particulate composite containing a fixed volume fraction of inclusions is predicted to be inversely proportional to its creep (or densification) viscosity.

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**Acknowledgement:** This work was supported by the Division of Materials Sciences, Office of Basic Energy Sciences, U.S. Department of Energy, under Contract No. DE-AC03-76SF00098. This work was performed in the Lawrence Berkeley Laboratory when Dr. M.N. Rahaman was Visiting Staff Scientist in the Materials and Chemical Sciences Division.



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## FIGURES

Fig. 1. Ratio of the densification strain rate to the creep strain rate versus relative density at a constant applied stress of 0.2 MPa. The numbers in parenthesis represent the relative green density and the sintering temperature ( $^{\circ}\text{C}$ ).

Fig. 2. Axial strain versus temperature for ZnO powder compacts sintered at a constant heating rate of  $4^{\circ}\text{C}/\text{min}$  and under the uniaxial stresses shown.

Fig. 3. Axial strain versus radial strain for ZnO.

Fig. 4. Axial strain versus radial strain for  $\text{Al}_2\text{O}_3$ . (A) Heating rate,  $4^{\circ}\text{C}/\text{min}$ ; applied load, 7 N, (B) Isothermal,  $1350^{\circ}\text{C}$ ; applied load, 7 N, and (C) Heating rate,  $4^{\circ}\text{C}/\text{min}$ .; applied load, 0 N.

Fig. 5. Various strains in a densifying and creeping compact.

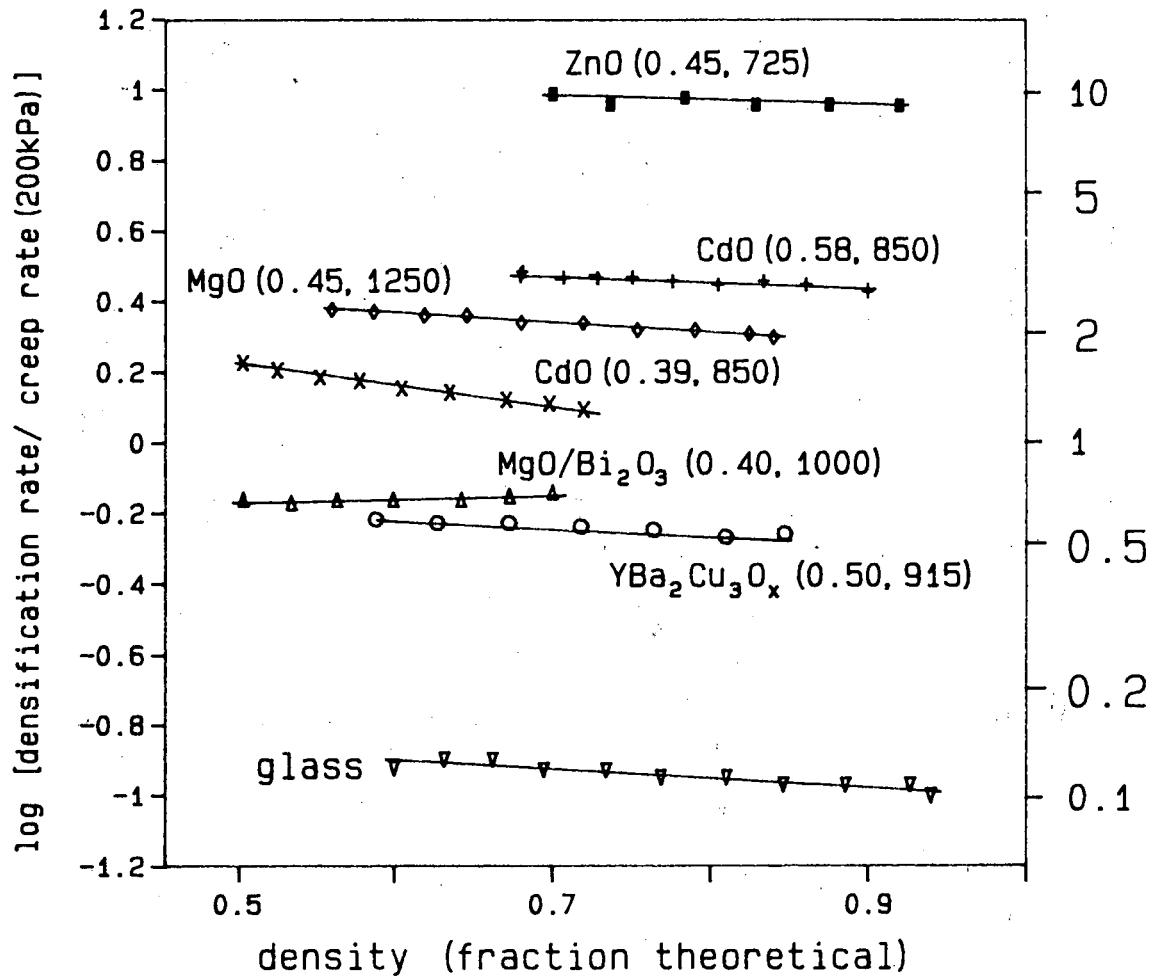
Fig. 6. Relative density,  $\rho$ , and creep strain,  $\epsilon_c$ , calculated from Figs. 2 and 3 and Eqs. (6) and (7).

Fig. 7. (a) Axial strain versus radial strain for ZnO with constant density line. (b) Hypothetical construction of the axial strain versus radial strain from  $\epsilon_z$  (measured from sample), isotropic line, and constant density line.

Fig. 8. Corrections of axial and radial strain from constant load ( $\epsilon_z, \epsilon_r$ ) to constant stress ( $\epsilon_z', \epsilon_r'$ ).

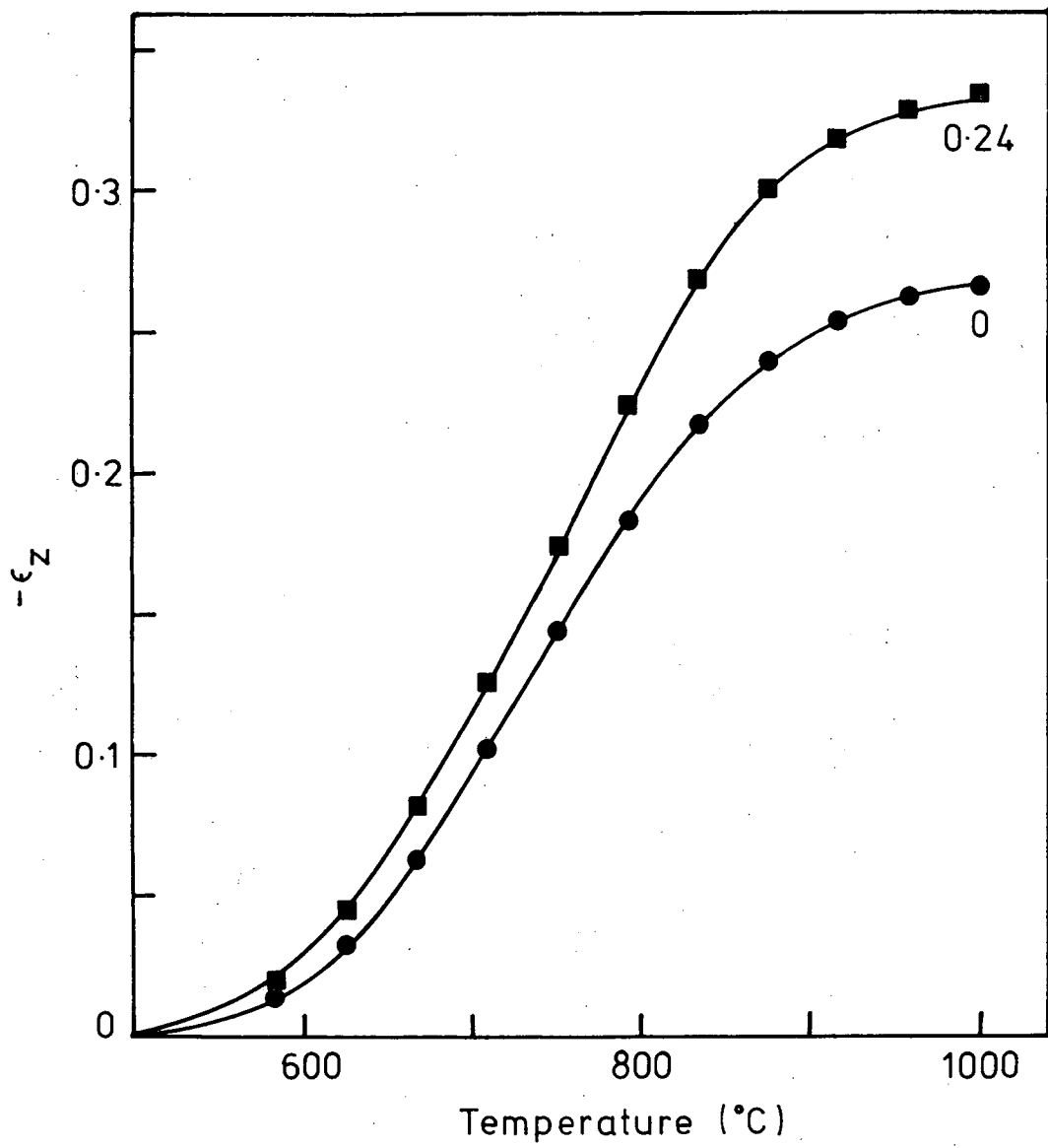
Fig. 9. Axial strain versus radial strain for ZnO. Constant load at 7 N (uncorrected), constant stress at .24 MPa (corrected), 0 load (isotropic).

Fig. 10. (a) Ratio of the densification to the creep strain rate versus temperature for the uniaxial stress shown. (b) Relative densification rate,  $\dot{\rho}/\rho$ , and creep strain rate,  $\dot{\epsilon}_c$ , versus temperature at the constant uniaxial stress shown.



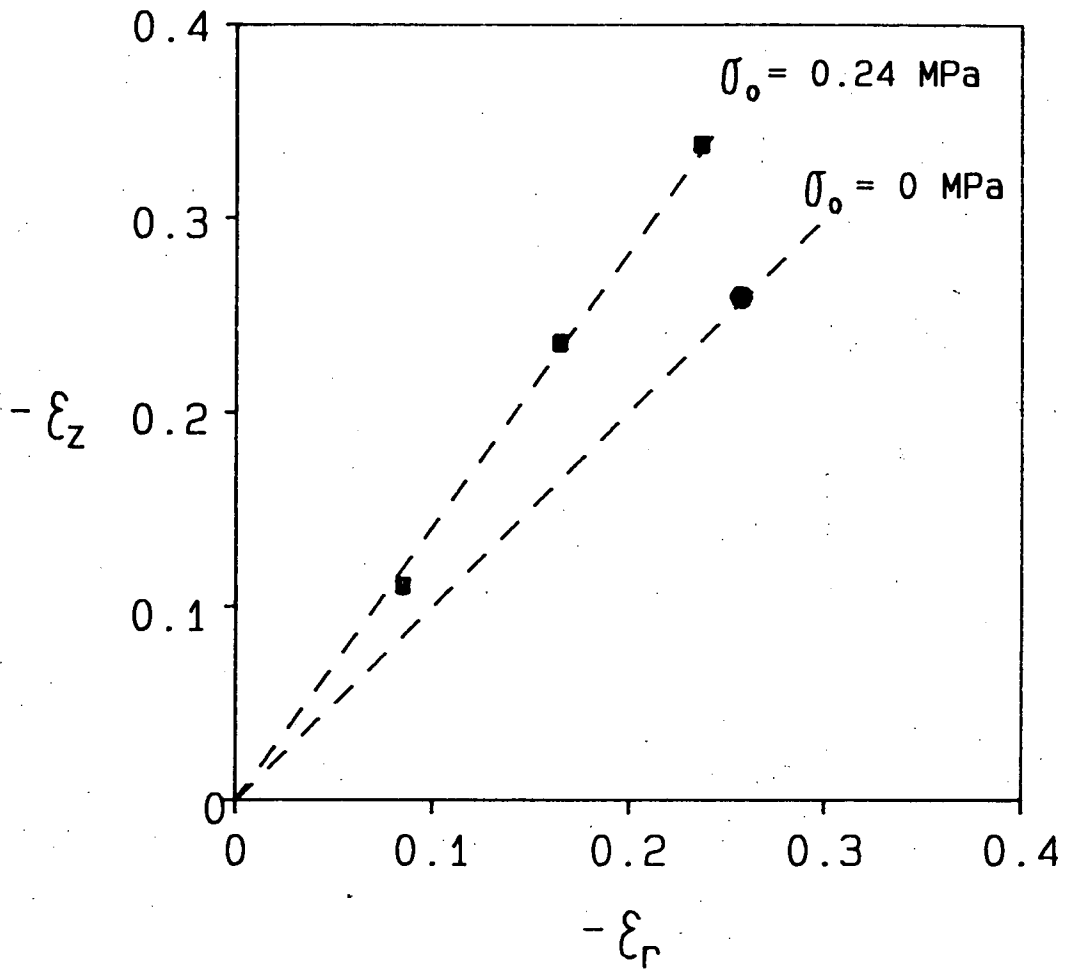
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Fig. 1



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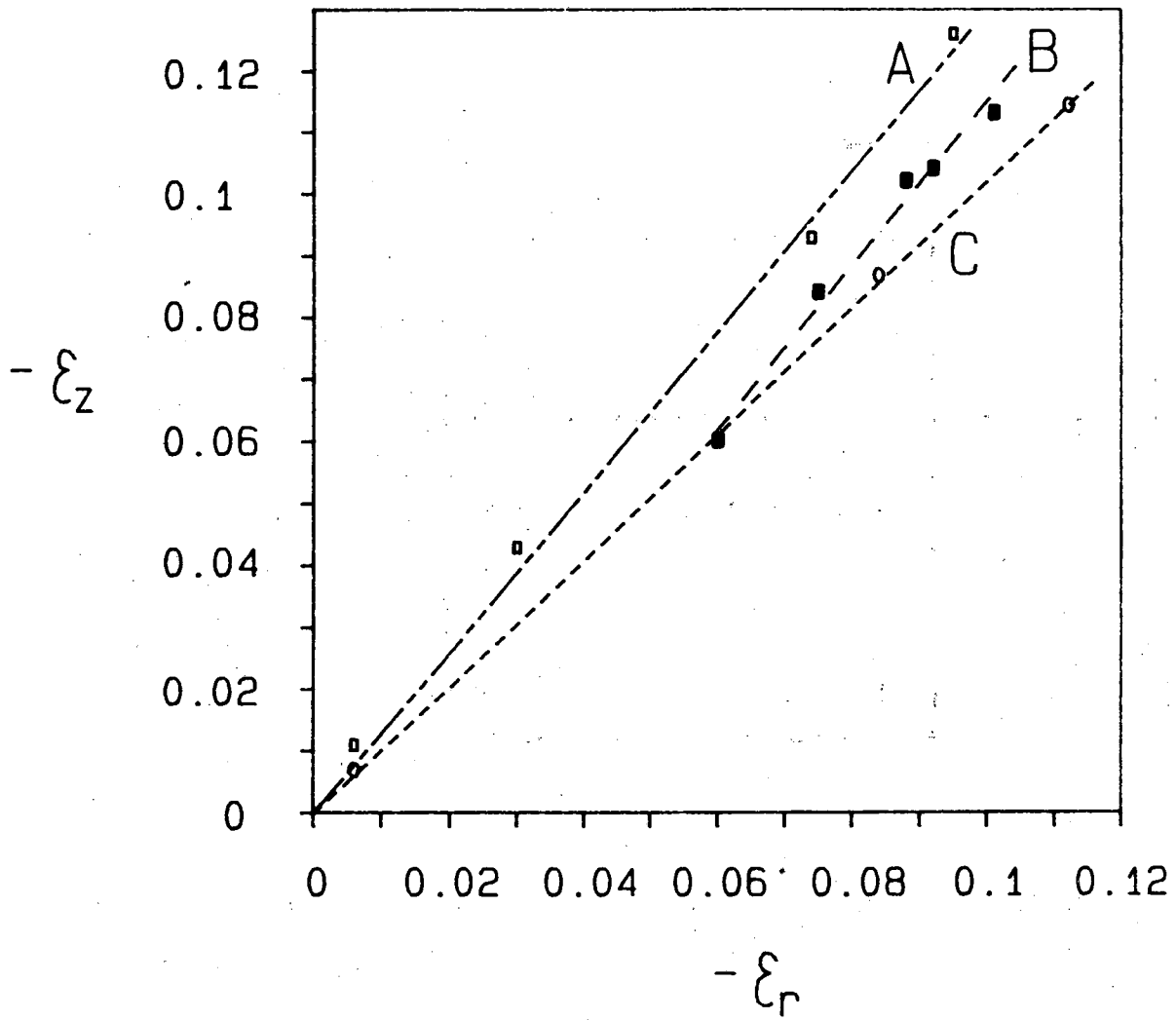
Fig. 2



XBL 886-2025

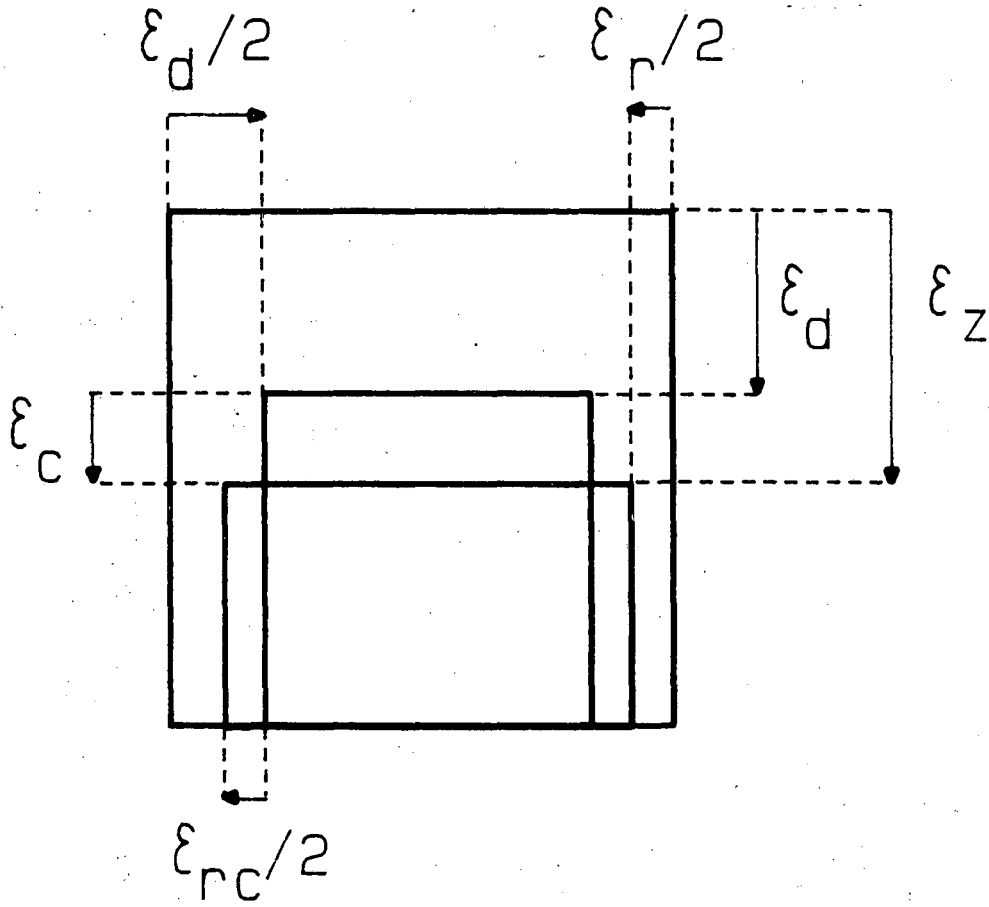
Fig. 3





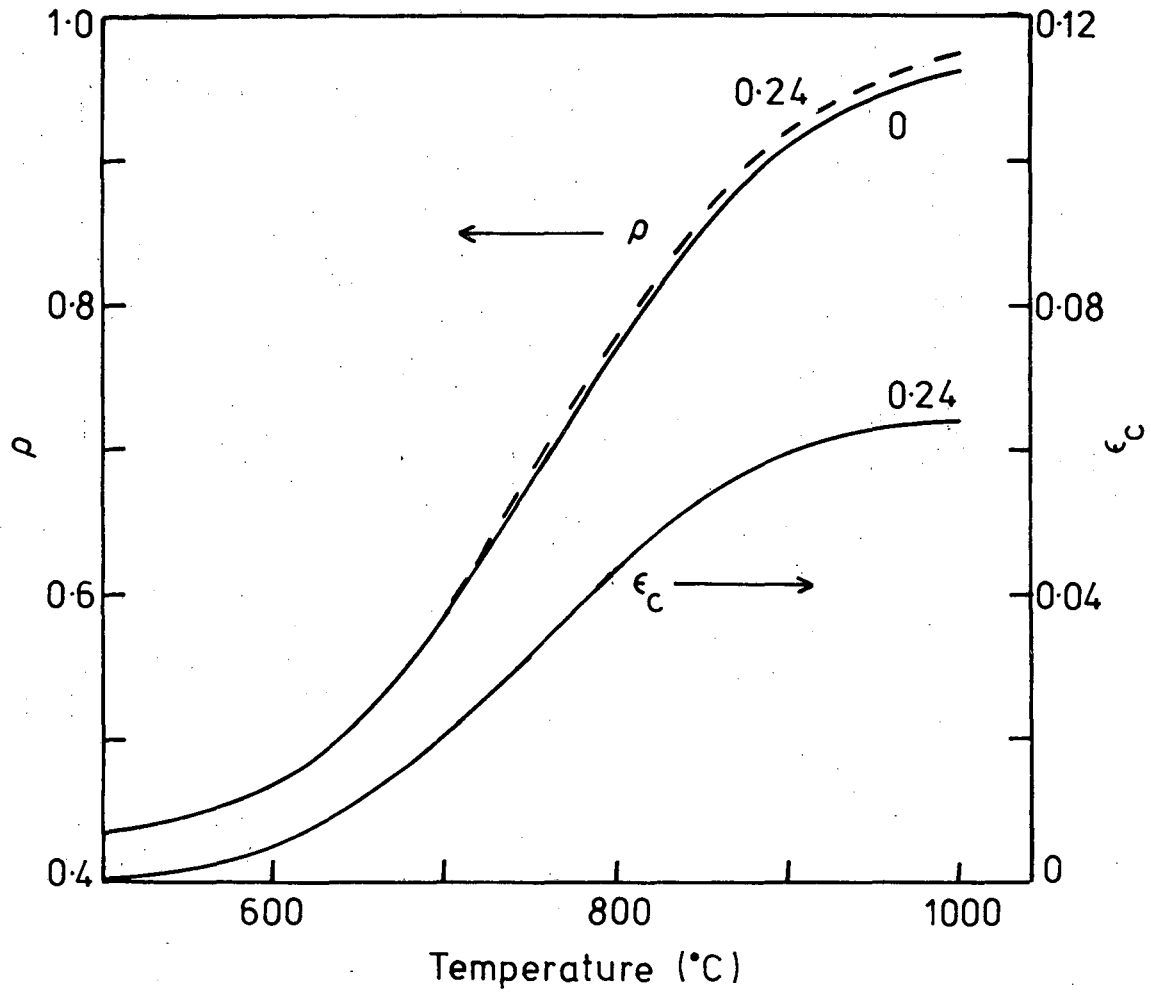
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Fig. 4



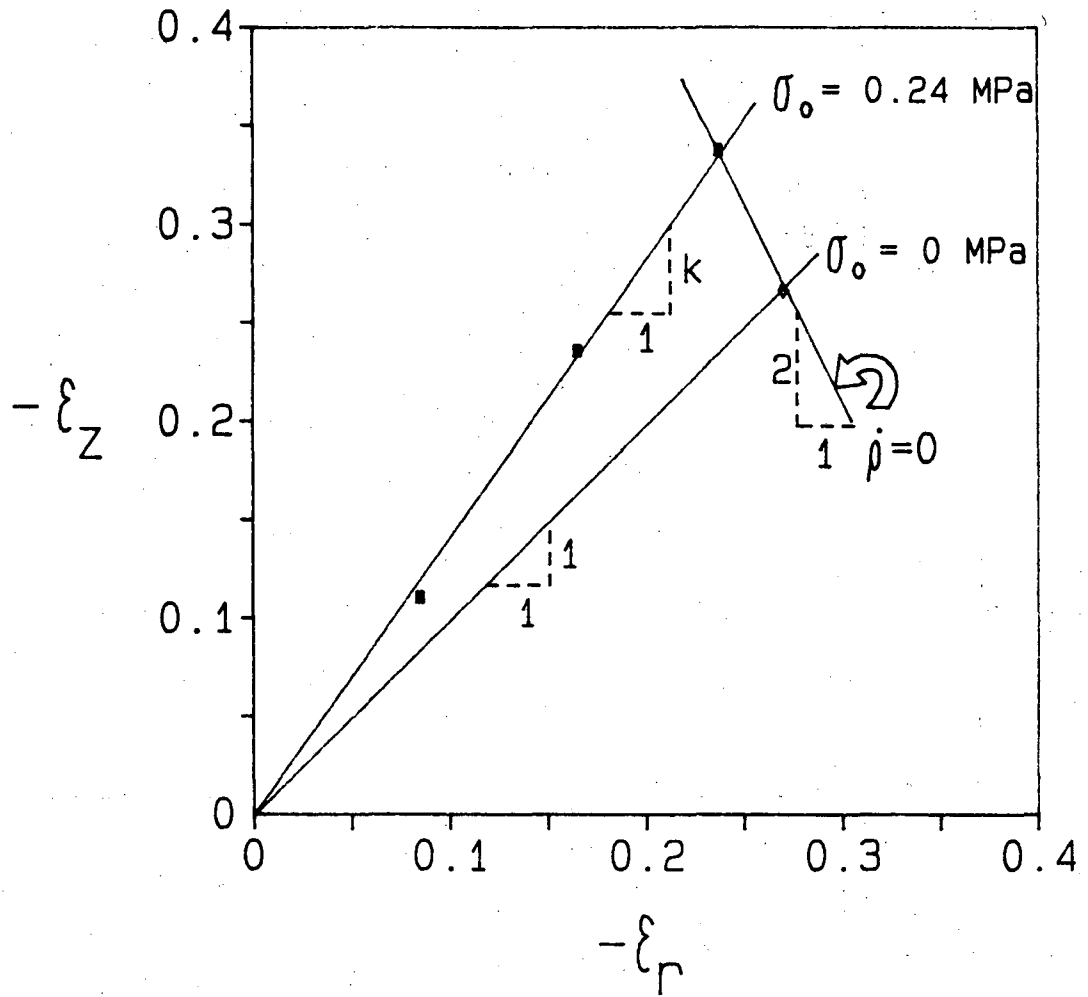
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Fig. 5



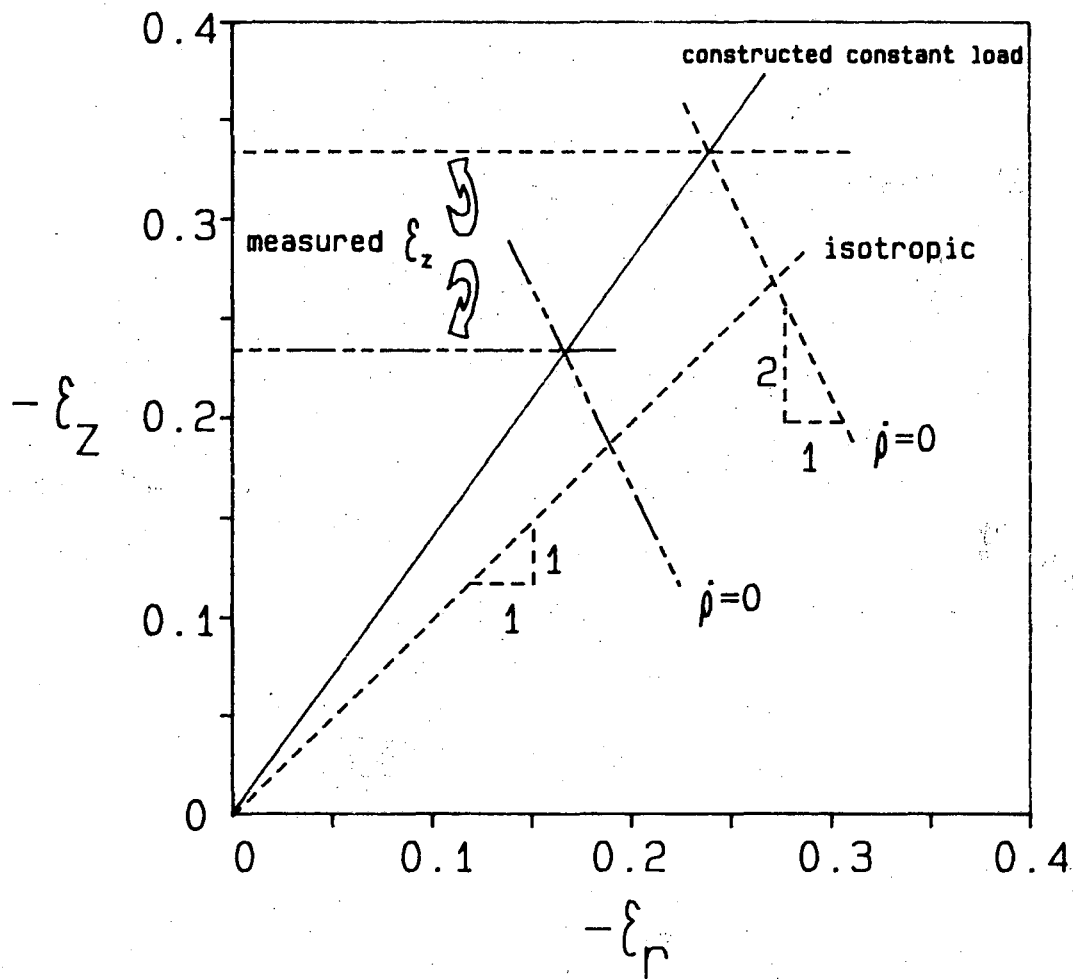
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Fig. 6



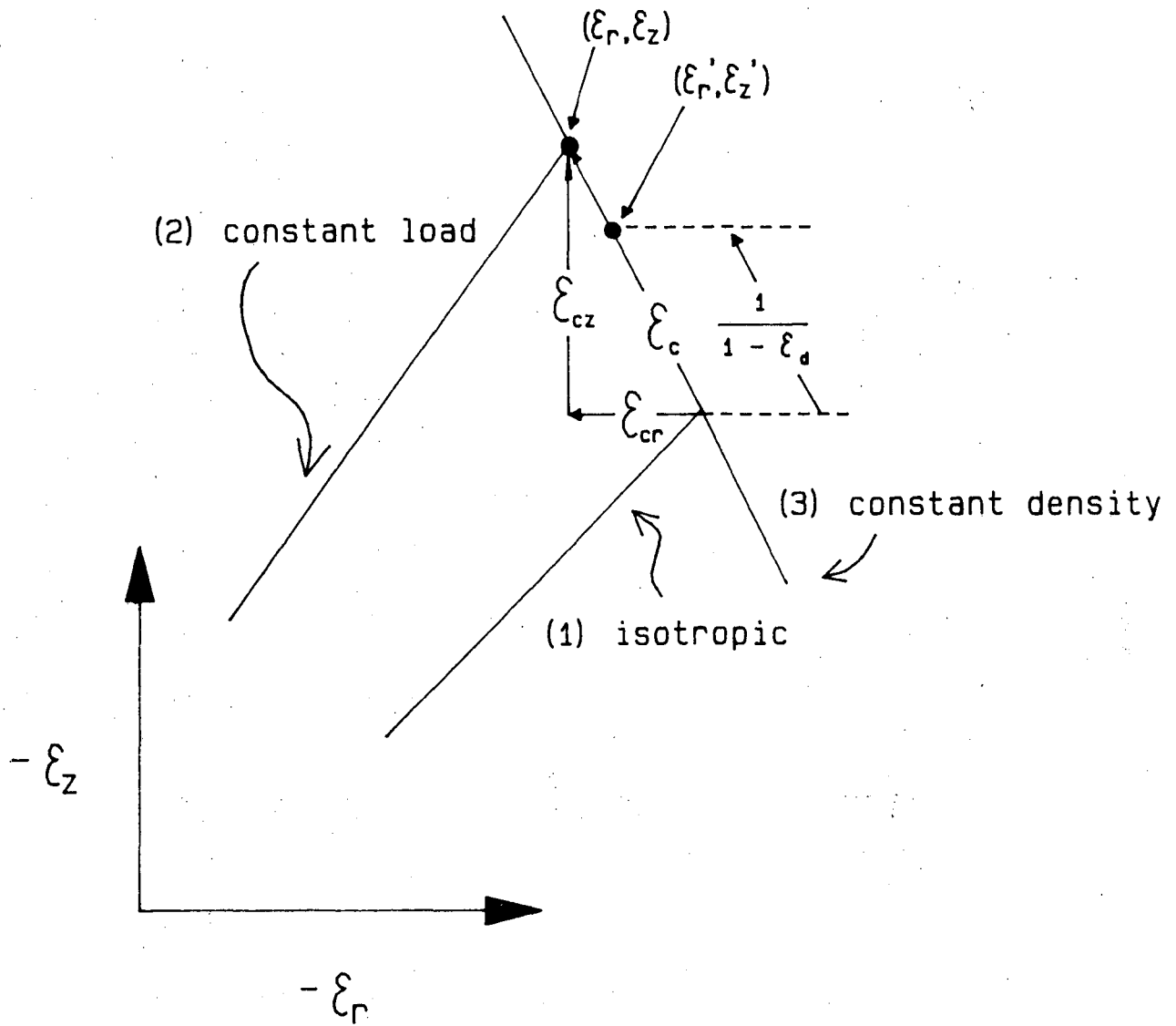
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Fig. 7a



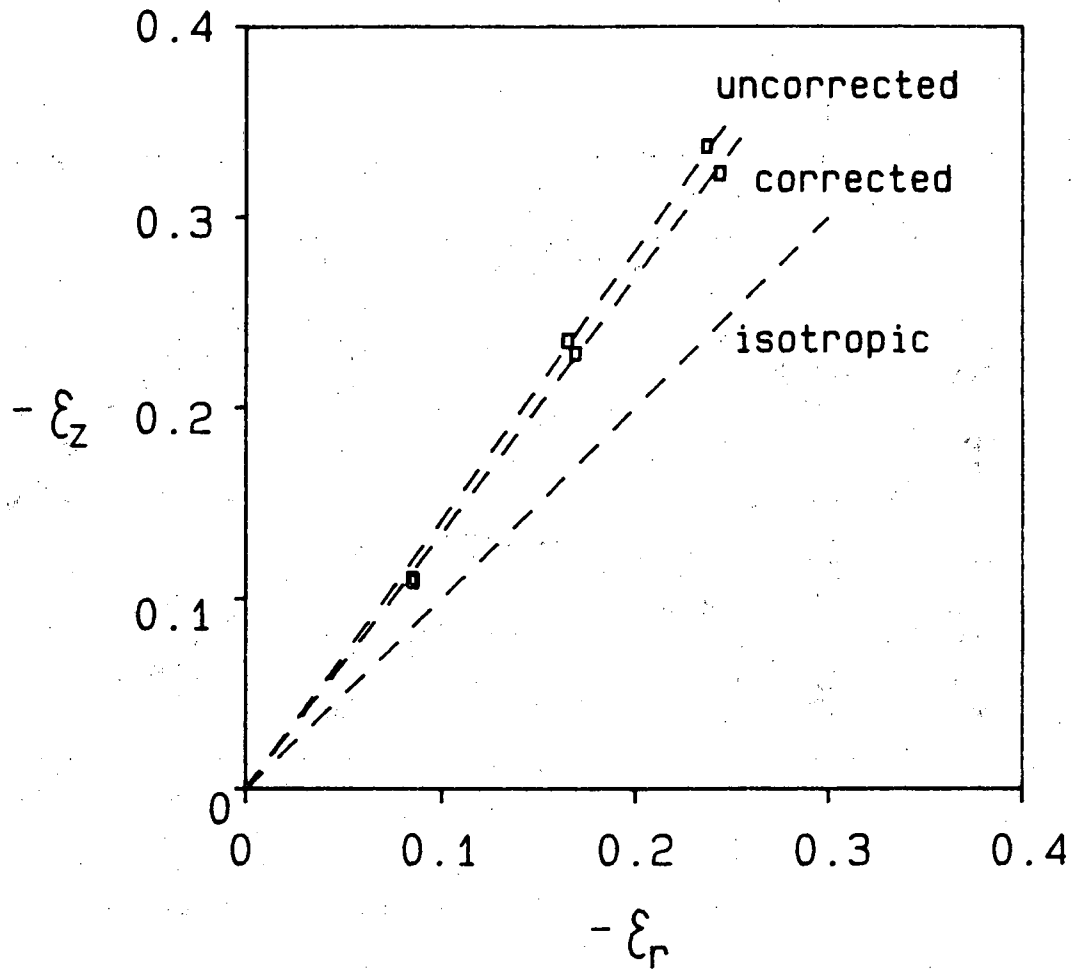
XBL 886-2029

Fig. 7b



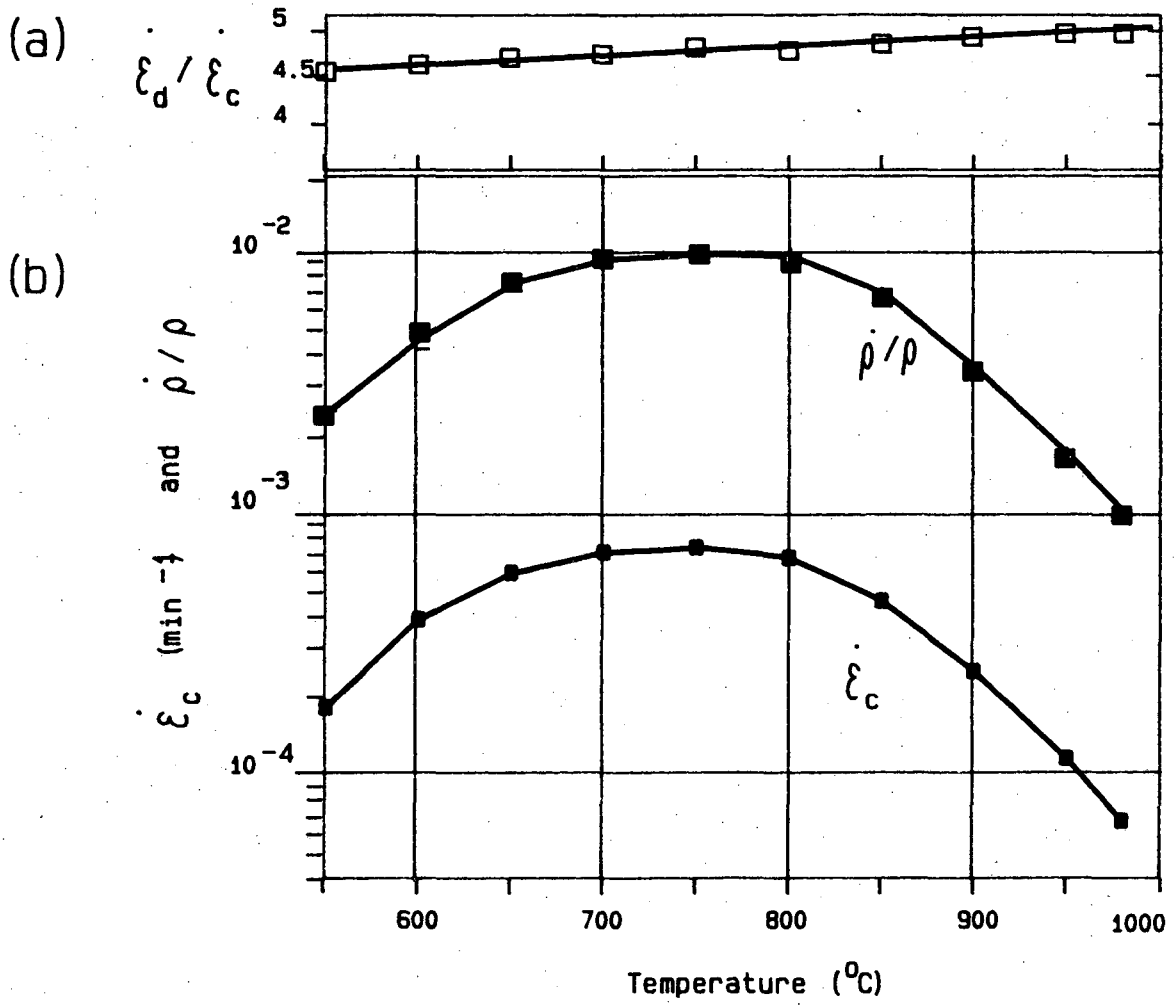
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Fig. 8



XBL 886-2031

Fig. 9



XBL 886-2032

Fig. 10



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