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M.N. Rahaman, L.C. De Jonghe
and C.H. Hsueh

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Lawrence Berkeley Laboratory
University of California
Berkeley, California 94720

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CREEP DURING SINTERING OF POROUS COMPACTS

M. N. Rahaman, L. C. De Jonghe and C.H. Hsueh
Center for Advanced Materials, Lawrence Berkeley Laboratory
and
Department of Materials Science and Mineral Engineering
University of California, Berkeley
Berkeley, CA 94720

Abstract

Creep during sintering of powder compacts of cadmium oxide was studied in a loading dilatometer by applying a small, transient, uniaxial load to the compacts. After removal of the load, the axial shrinkage rate is lower but the radial shrinkage rate is actually higher than that of a compact sintered under no load. This reduction in the axial shrinkage rate is more pronounced for longer, transient loading times. The results provide further support for a mechanism of simultaneous creep and densification in which creep at constant volume occurs by diffusion controlled grain boundary sliding.

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I. INTRODUCTION

It is now widely recognized that inhomogeneities within a powder compact can lead to non-uniform sintering rates which, in turn, produce transient (and sometimes residual) stresses.[1-5] These stresses may influence creep processes within the porous sintering compact. It is therefore important to quantify the interaction of creep and densification processes during sintering.

In previous work by Rahaman and De Jonghe[6,7] the usefulness of a loading dilatometer to study simultaneous creep and densification was demonstrated. In the loading dilatometer, a small, measured, uniaxial load is applied to the sintering compact. This small load does not affect the volumetric densification rate but may cause extensive creep. A later paper,[8] extending the study in both theoretical and experimental areas, showed how a number of important sintering parameters such as the sintering stress, the effective viscosity and the sintering mechanism, could be measured. Based on these results and some initial results involving transient loads, a simplified model was put forward to interpret the microstructural processes occuring during simultaneous creep and densification. In this model, creep at constant volume occurred by a mechanism of grain boundary diffusion controlled grain boundary sliding.

The purpose of this paper is to report the results of further exeriments performed to test the validity of this microstructural model, and to compare them with theoretical calculations based on this model.

II. EXPERIMENTAL PROCEDURE

Cadmium oxide powder* was uniaxially pressed at 20MPa into cylindrical compacts (6mm by 6mm) and then isostatically pressed at ~60MPa to give compacts with a green density of 0.56 ± 0.01 of theoretical. All sintering experiments, with or without load, were performed at 1123K in flowing air (~$50cm^3$/min) in a loading dilatometer described in detail elsewhere.[5]

In one set of experiments, different compacts were sintered under no load and under a constant load of 5N for two hours. In another set of experiments involving transient loads, compacts were sintered under 5N load for 11 min and 55 min, respectively, and then the load was removed. Continued sintering under no load then occurred, and the experiments were terminated after a total time of two hours. Finally, the (differential) radial shrinkage was measured after the removal of the transient load as follows: Two compacts, one subjected to 5N load in the axial direction and the other subjected to no load, were sintered for 25 min and the differential axial shrinkage was measured. The compacts were removed and flat and parallel surfaces were machined on their cylindrical surfaces, to allow for shrinkage measurements in the radial direction. These compacts were sintered further, both subjected to no load, and the differential radial shrinkage was measured.

*Reagent grade, J.T. Baker Chemical Company, Phillipsburg, N.J.

The density of the compacts at any time was calculated from the green density and the measured shrinkage. The final density was also measured using Archimedes'principle.

III. RESULTS AND DISCUSSION

Fig. 1 shows the axial shrinkage, $\Delta L/L_o$, versus time, t, for compacts sintered under no load (Curve A), at a constant load of 5N (B) and under transient loads of 5N (C and D). For Curves C and D the load was quickly removed after times, t^*, of 11 and 55 min, respectively. (L_o = intial sample length, and $\Delta L = L-L_o$, where L = instantaneous sample length). A load of 5N represents a stress of 0.22 MPa and t = 0 represents the beginning of sintering. The sintering temperature was reached after t = 8 min and each curve is reproducible to within $\pm 2\%$.

As found earlier,[7] the small applied stresses have almost no effect on the volumetric densification rate. The density of all the compacts at any sintering time agreed to within $\pm 1\%$. The final density (0.86 of theoretical) calculated from the dimensional changes was also in good agreement with that measured using Archimedes' principle.

The axial shrinkage rate, $(\frac{1}{L_o}\frac{dL}{dt})$, is plotted versus t in Fig. 2. The letter by each curve represents the same conditions as stated for Fig. 1. It is seen that after the removal of the transient load, the axial shrinkage rate (Curves C and D) is lower than that of the compact sintered throughout under no load (Curve A). Moreover this reduction depends on the time, $t,^*$ at which the transient load was removed, being more pronounced at longer t^*.

Fig. 3 shows the differential shrinkage in the radial direction between two compacts, one previously sintered for 25 min under 5N load in the axial direction and the other sintered under no load for the same time. These results show that the previously loaded compact shrinks faster in the radial direction after the load is removed.

It is important now to compare the nature of these results with those expected from the simplified microstructural model put forward earlier[8] to account for simultaneous creep and densification in CdO. Fig. 4 shows the model in which the grain boundaries in a porous compact are subjected to an external, uniaxial stress. Depending on their orientation, boundaries may be under compression, under tension or sliding. For CdO subjected to low stresses, the rate controlling creep and densification mechanisms are the same, namely, grain boundary diffusion.[8] Creep at constant volume is controlled by diffusion of matter from boundaries under compression into neighboring pores (A), and from neighboring pores (B) into boundaries under tension. According to this model, a CdO compact will behave as it were denser in the direction of the applied stress. Thus after removal of the stress, the axial shrinkage rate should be lower than that of a compact sintered throughout under no stress. Moreover, since the effect of load on the microstructure is cumulative, this decrease in the axial shrinkage rate should be more pronounced after longer transient loading times, t^*. These predictions are very well substantiated by the results of Fig. 2.

On the basis of the microstructural model, a quantitative estimate may be made of the apparent densification rate in the axial direction (as measured by the dilatometer) after application or removal of the transient loads.

Consider a compact undergoing sintering under no load. Then for CdO the densification rate at time $t = t^*$, say, may be written as [8]

$$\dot{\rho}_o = K \Sigma_a \phi_a^{3/2} \qquad \text{Eqn. (1)}$$

where K is a kinetic constant that includes the grain size, Σ_a is the sintering stress and ϕ_a is the stress intensification factor.

If a stress, σ, is applied to a freely sintering compact at $t = t^*$, then at $t^* + \delta t$, where δt is small, the apparent densification rate as measured by the dilatometer is given by

$$\dot{\rho}_\sigma = K(\Sigma_a + \sigma\phi_a)\, \phi_a^{3/2} \qquad \text{Eqn. (2)}$$

Then

$$\dot{\rho}_\sigma/\dot{\rho}_o = 1 + \sigma\phi_a/\Sigma_a \qquad \text{Eqn. (3)}$$

Both ϕ_a and Σ_a can be obtained from experiment.[8]

Consider now a compact undergoing sintering under a constant stress, σ. Then the apparent densification rate at $t = t^*$ is given by

$$\dot{\rho}'_\sigma = K\, (\Sigma_b + \sigma\phi_b)\, \phi_b^{3/2} \qquad \text{Eqn. (4)}$$

where Σ_b and ϕ_b are the appropriate sintering stress and stress intensification factor, respectively. Σ_b and ϕ_b are somewhat different from Σ_a and ϕ_a in Eqn. 1 since the apparent density as measured by the dilatometer is higher in sintering under load, and Σ_b and ϕ_b are functions of density.

If the stress is now quickly removed from a compact at $t = t^*$, then at $t^* + \delta t$, the apparent densification rate of this unloaded compact is given by

$$\dot{\rho}_o = K \Sigma_b \phi_b^{3/2} \qquad \text{Eqn. (5)}$$

Then

$$\dot{\rho}_\sigma'/\dot{\rho}_o' = 1 + \sigma\phi_b/\Sigma_b \qquad \text{Eqn. (6)}$$

And

$$\dot{\rho}_\sigma'/\dot{\rho}_o = (\Sigma_b/\Sigma_a)(\phi_b/\phi_a)^{3/2} \qquad \text{Eqn. (7)}$$

Σ_b and ϕ_b are derived, first by using the axial shrinkage (Fig. 1) to calculated the apparent density, as measured by the dilatometer, and then found at this density from other experiments that determined Σ and ϕ as a function of density.[8] The values calculated for $\dot{\rho}_\sigma/\dot{\rho}_o$, $\dot{\rho}_\sigma'/\dot{\rho}_o'$ and $\dot{\rho}_\sigma'/\dot{\rho}_o$ using equations 3, 6 and 7 are in good agreement with the experimental results (extrapolated to $t = t^*$) to within $\pm 2\%$, as is evident from Table I.

Table I. Comparison of the experimental results with theoretical results calculated on the basis of the microstructural model.

RATIO	EXPT.	THEORY	COMMENT
$\dot{\rho}_o'/\dot{\rho}_o$	0.89	0.88	Curve C, $t^* = 11$ min.
$\dot{\rho}_o'/\dot{\rho}_o$	0.74	0.74	Curve D, $t^* = 55$ min.
$\dot{\rho}_\sigma'/\dot{\rho}_o$	1.60	1.58	Curve A, $t^* = 20$ min. (Fig. 10, Reference 8)

IV CONCLUSIONS

The small applied stresses cause constant volume creep without affecting the volumetric densification rate. Another consequence of the microstructural model is that following the removal of the stress, the radial shrinkage should be less than that of a compact sintered throughout under no load, i.e. the compact sintered under the uniaxial stress appears denser in the axial direction but less dense in the radial direction compared to a compact sintered under no load. The results of Fig. 3 clearly confirm this prediction.

It is clear that the mechanism of constant volume creep by diffusion controlled grain boundary sliding, put forward earlier to explain the microstructural changes occurring during simultaneous creep and densification of CdO, has been further supported by the present results. It should be stressed that the model has been verified so far only for the case in which the creep process is grain boundary diffusion controlled grain boundary sliding. The study of simultaneous creep and densification in other ceramic systems is currently in progress.

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1. Axial shrinkage versus time for compacts sintered under no load (Curve A), a constant load of 5N (B) and transient loads of 5N (C and D).

2. Axial shrinkage rate versus time.

3. Differential radial shrinkage, after removal of the axial load, versus time.

4. Microstructural model showing pores and grain boundaries subjected to an externally applied load. (Reference 8).

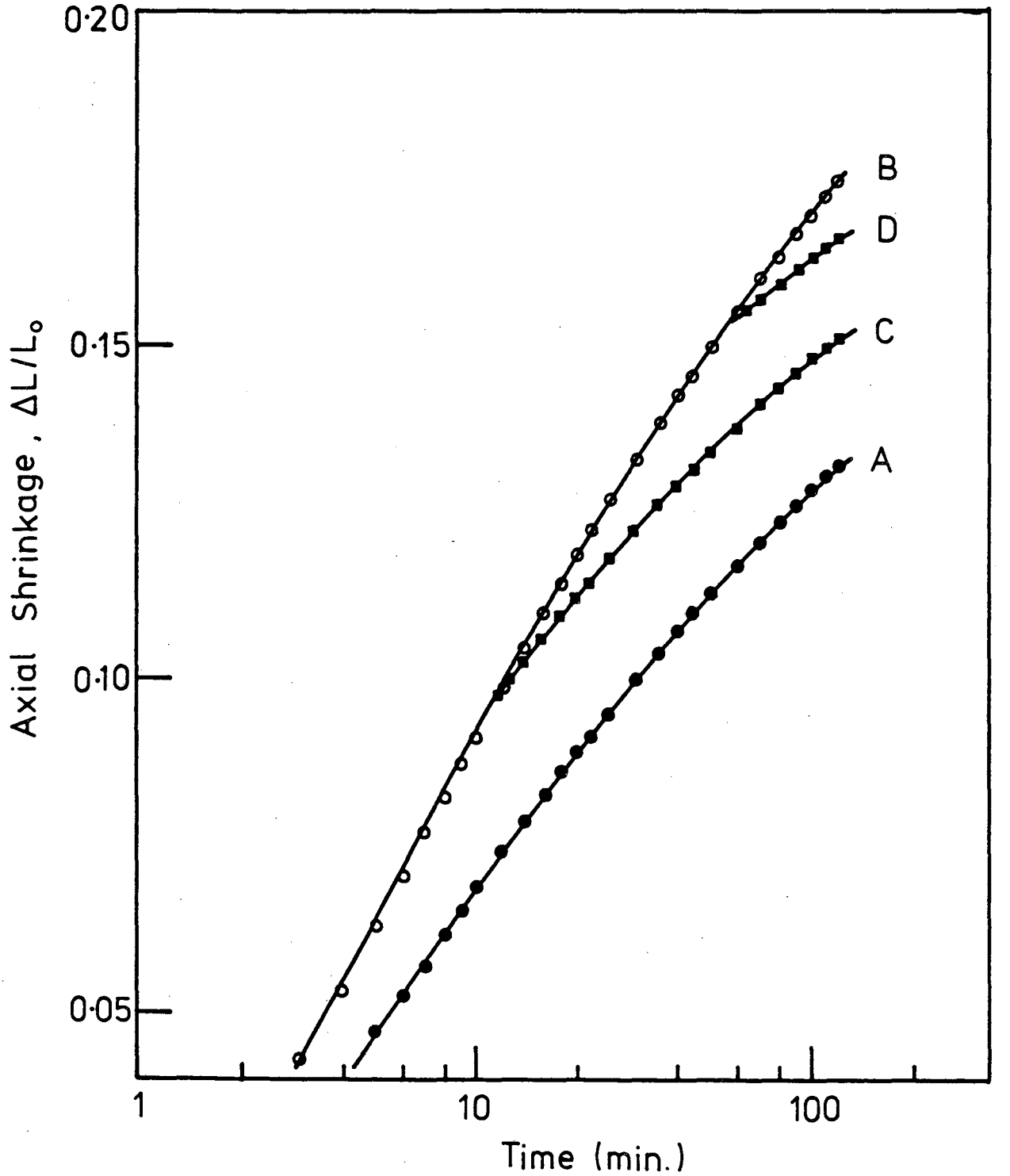

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Figure 1

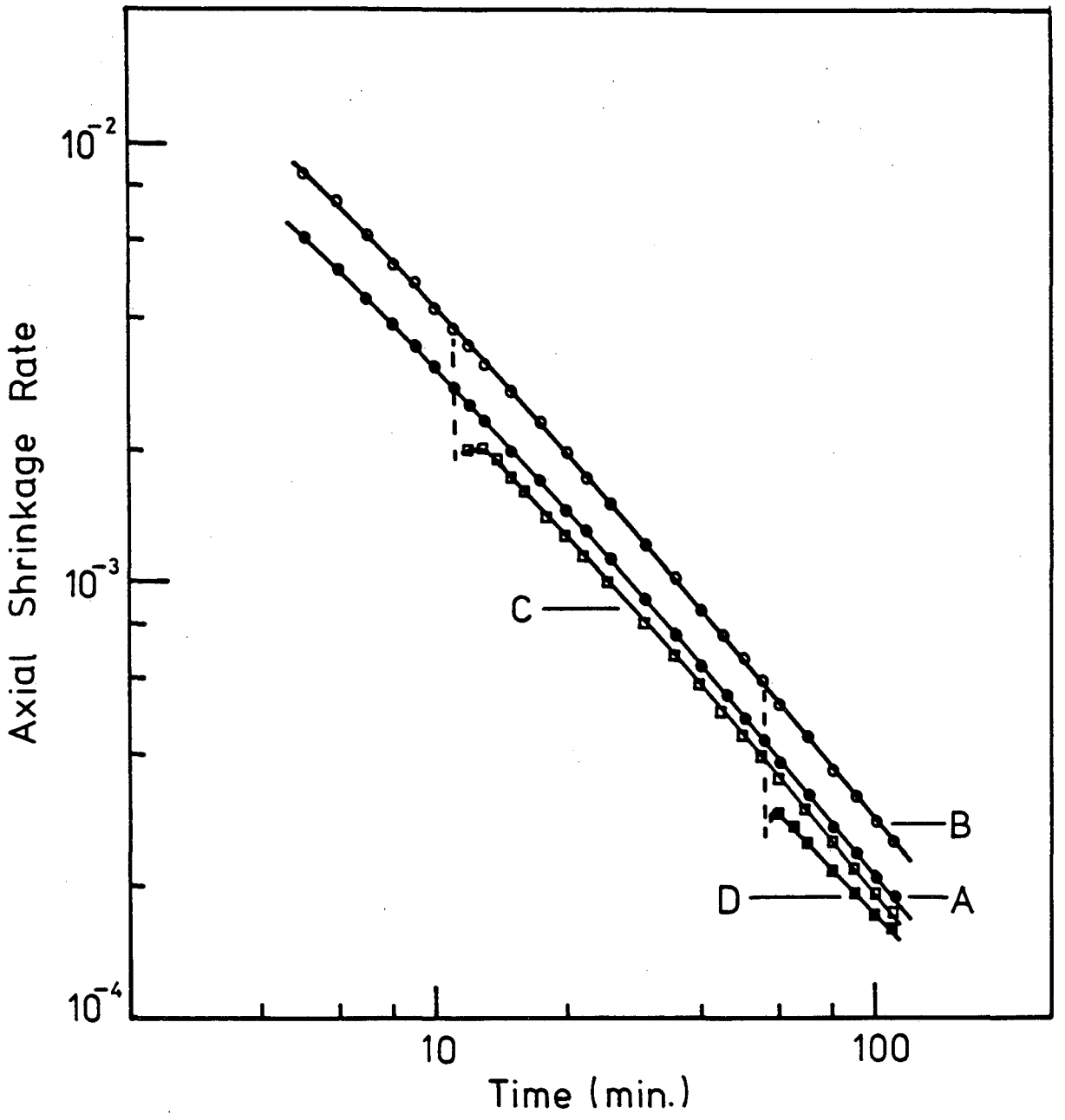

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Figure 2

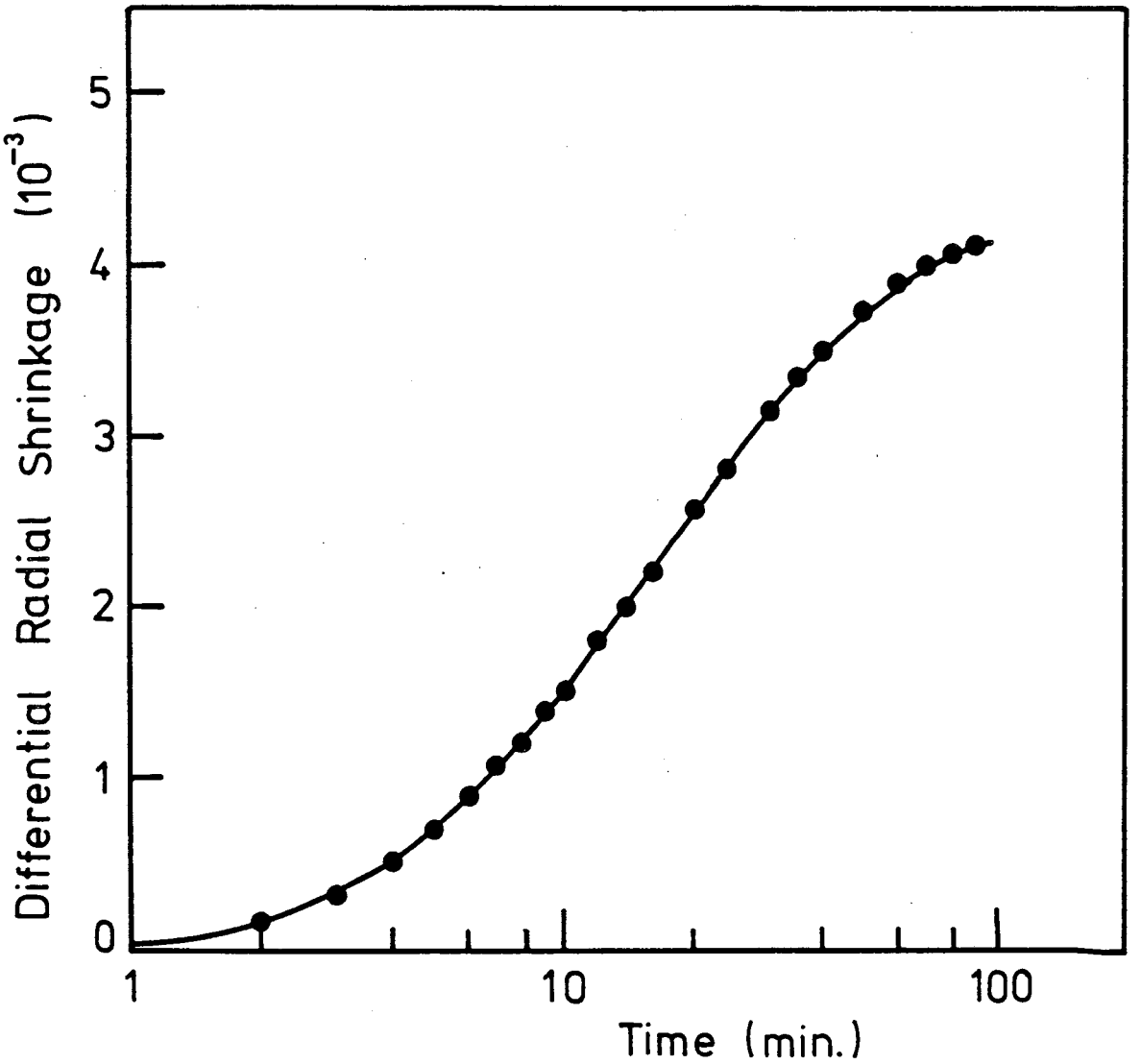

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Figure 3

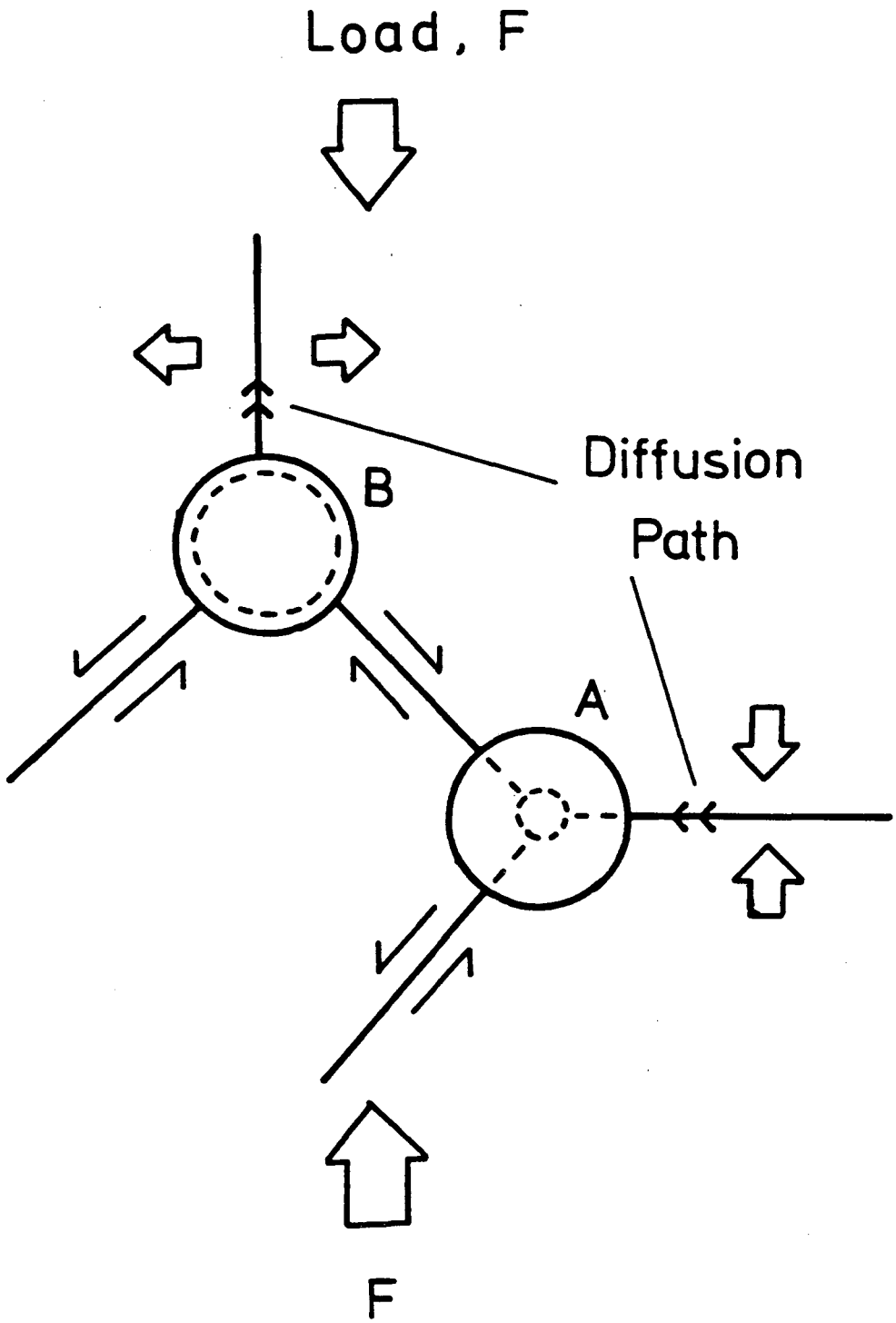

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Figure 4

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