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A LOADING DILATOMETER

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Center for Advanced Materials

DISCLAIMER

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ABSTRACT

A loading dilatometer has been constructed to study the sintering behaviour of powder compacts subjected to low uniaxial stresses between 0 and 0.4 MPa and at temperatures up to 1250K. A novel feature of the instrument is a device for the application of a controlled and measured stress to the sintering compact. Constant, as well as transient stresses can be applied and the atmosphere inside the dilatometer can be controlled. Some representative results are reported.

1. INTRODUCTION

Ceramic materials and some metals are fabricated by compacting powders to form a "green body, followed by sintering[1] at high temperatures. Powder characteristics and compacting procedures cause the formation of inhomogeneities within the green body. These inhomogeneities,[2,3,4] such as agglomerates and density variations, give rise to differential sintering rates and stresses within the body during sintering and may even lead to crack-like voids in the sintered material. The measurement and calculation of these stresses at a microscopic level is very difficult. It is hoped that by addressing the problem experimentally, at a macroscopic level, some progress can be made.

This paper describes a loading dilatometer which has been constructed by modifying a Harrop[5] differential dilatometer, to study the sintering behaviour of powder compacts subjected to low uniaxial stresses between 0 and 0.4 MPa and at temperatures up to 1250K. The atmosphere inside the dilatometer can be controlled. In practice, the sintering of a single compact or the difference in sintering between two compacts can be studied. A novel feature of the dilatometer is device for the application of a controlled and measured stress to the sintering compact. Stresses can be applied and removed quickly, so that experiments under constant as well as intermittent stresses can be performed. One advantage of the sintering technique described here is that inaccuracies due to die-wall friction, as in hot-pressing[1] at low stresses do not occur. There is a small amount of friction at the

contact surfaces of the powder compacts but the effect is not significant. In addition to investigating the problems of stresses arising from differential sintering rates in powder compacts, the loading dilatometer experiments may also be used more generally, for a further understanding of the sintering process.

2. DESCRIPTION OF THE APPARATUS

A detailed illustration of the loading dilatometer is shown in fig. 1 [a]. A schematic diagram, viewed from above the instrument, is shown in fig. [b]. In the following, the numbered references are made to both figures. A few references have been omitted from fig 1 [b] because of the schematic nature of this diagram.

In general, the dilatometer is attached to a steel platform 2, which moves smoothly on fixed horizontal rails. The platform is connected to an electrically driven motor that allows the dilatometer to be moved at a known rate towards, or away from, a horizontal tube furnace.

The inner quartz tube 12 (35 mm diameter and 350 mm long) is fixed onto an aluminum base 21, and serves primarily to support the two powder compacts 13, 15 and the quartz push rods 18, 19. Part of it has been cut away as illustrated, to allow the setting up of the compacts and removal of the push rods, when necessary. The quartz push rods (6.5 mm diameter and 450 mm long) pass through holes in a low friction, boron nitride block 17, which helps to keep the rods in position. The powder compacts (usually 6 mm diameter and 6 mm long) are separted from the base of the inner quartz tube and the push rods by quartz spacers

14, 16. The junction of a Pt/Pt 13% Rh thermocouple is directly under the compacts and allows the temperature to be followed on a digital indicator and a recorder. The outer quartz tube 11 (45 mm diameter and 400 mm long) is attached to the lauminum base 21 using screws, with a rubber seal providing an air-tight contact. The atmosphere inside the dilatometer can be controlled by passing a gas through the inlet 20 and out through the outlet 10.

An air-tight perspex box 1 (300 mm long by 70 mm by 90 mm) encloses the transducer and strain gauge system. It is held firmly onto the aluminum base using a rubber seal and metal clips. A stainless steel rod 4 (2-5 cm diameter by 10 cm) rigidly attached to the base 21, is used to support the transducer and strain gauge system. A micrometer 3, allows careful and precise movement of the transducer and strain gauge systems relative to the steel rod 4.

The transducer and strain gauge systems will now be described. The push rods are attached to two aluminum blocks 8, 23, suspended from the rigid aluminum support 22 by means of low tension springs 6. Since one block 8, contains the coil of a linear voltage displacement transducer,[7] while the other block 23, carries the magnet 5 of the transducer, ralative movement of the push rods causes a change in output of the transducer. A transducer amplifier [8] and a recorder are used to record the movement (due to the shrinkage of the powder compacts), provided the system has been calibrated. The transducer magenet is attached to the aluminum plate 24 by means of a screw thread. The initial reading of the transducer can then be easily

adjusted by screwing the magnet slightly, forwards or backwards, relative to the coil. This is very convenient, especially when the powder compacts differ slightly in length. The use of the low tension springs to support the blocks 8, 23 ensures that only a very small load is applied to the compacts by the transducer system.

The load on one compact is supplied by means of compressed gas acting against a piston. A sketch of the pressure chamber 28 and the piston 26 is shown in fig 2 [a]. The pressure chamber consists of a steel cylinder, 20 mm diameter and 60 mm long, the ends of which are in contact with rubber seals. Friction between the piston head and the cylinder is minimized by allowing the gas to flow around the O-ring attached to the piston head, and through a controlled leak 27. In operation, gas from a nitrogen cylinder is delivered at a constant pressure of about 0.2 MPa to two pressure regulators,[9] the outlet pressure of which can be adjusted to apply the required load to the specimen. The compressed gas forces the piston and the strain gauge[7] 25, attached to the piston, against a metal plate 24. The load is transmitted via block 23 and push rod 18 to compact 15. Loads can be applied or removed very quickly by opening or closing appropriate pin-head valves.

A sketch of the strain gauge is shown in fig 2 [b]. The gauge consists of two L - shaped blocks of aluminum connected by a central strip. Attached to this strip are four constantan foils wired to form a Wheatson bridge circuit. The resistance of the circuit changes as the foils are strained. The applied load is measured using a digital

strain indicator,[10] calibrated by easily removing the strain gauge and applying known loads to it.

Generally, the instrument described can be used to investigate a number of effects, the more important of which are mentioned here. First, by replacing compact 13 with a fully dense, reference pellet, the effect of small loads on the sintering of a powder compact 15 can be studied. Second, using two powder compacts, the effect of small loads on the differential sintering between the two compacts can be investigated. Third, the effect of small, transient or intermittent loads can be studied. In addition, studies of the effects of sintering atmosphere and temperature on any of these three types of experiments can be performed.

3. METHOD OF USING THE APPARATUS.

After the outer quartz tube and the plastic box (see fig. 1 [a]) have been removed from the dilatometer, the specimens are put in position and held by the springs 6. The transducer and recorder are adjusted to their initial readings. The required load on one compact is then applied by adjusting the nitrogen gas pressure using the pressure regulators. The load is quickley removed. The quartz tube and plastic box are carefully replaced, and atmosphere gas, flowing at a known rate (usually 50 cc/min) is passed into the dilatometer. After the furnace reaches the working temperature, the dilatometer is moved quickly and at a fixed rate, into it. The load on the specimen is then applied and the shrinkage and temperature, the dilatometer is moved quickly and at a fixed rate, into it. The load on the specimen is then

applied and the shrinkage and temperature of the compact recorded continuously. After sintering, usually for about 2 hours, the load is quickly removed and the dilatometer taken out of the furnace.

4. RESULTS

As an example, fig. 3 shows the results for the linear shrinkage versus time for CdO and ZnO compacts at 1123K and 973K, respectively, and subjected to low stresses between 0 and 0.3 MPa. The linear shrinkage is defined as the decrease in length divided by the initial length of the compact. All experiments were performed in flowing air. Each curve is reproducible to 2%. The dimensions of the compacts were measured before and after sintering, using a micrometer. The final shrinkage obtained using the micrometer was in excellent agreement with that obtained using the recording instruments. The stress on the compact can be maintained to within 5% over two hours. Experiments at 1123K and 0.25 MPa using a hot-pressed silicon nitride specimen showed that there was no creep in the quartz push rods.

Fig 4 shows an example in which the stress had been changed during the sintering of CdO at 1123K. The shrinkage rate is plotted versus log time. The stress was increased from 0 to 0.2 MPa after 20 minutes. For comparison, the results for sintering under constant stresses of 0 and 0.2MPa respectively, are also shown. After the stress change, the shrinkage rate increases rapidly before decreasing to a smooth variation with time. This feature is not due to mechanical instability of the instrument since previous experiments, using a hot-pressed silicon nitride specimen and high - sensitivity of the instruments, had

shown that the readings of shrinkage and stress stabilized to constant values in <5 sec. after the stress change.

A patent for this instrument has been filed.

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[6] Model 199, Omega Engineering, Stamford, Connecticut.

[7] Engineering and Technical Services, Lawrence Berkeley Laboratory, Berkeley, CA 94720.

[8] Model 300 C/60, Daytronic Corporation, Dayton, Ohio.

[9] Matheson Pressure Regulators, Models 70 and 70A.

[10] Model V/E - 20A, Instruments Division, Measurements Group, Raleigh, N. C.

LIST OF FIGURES

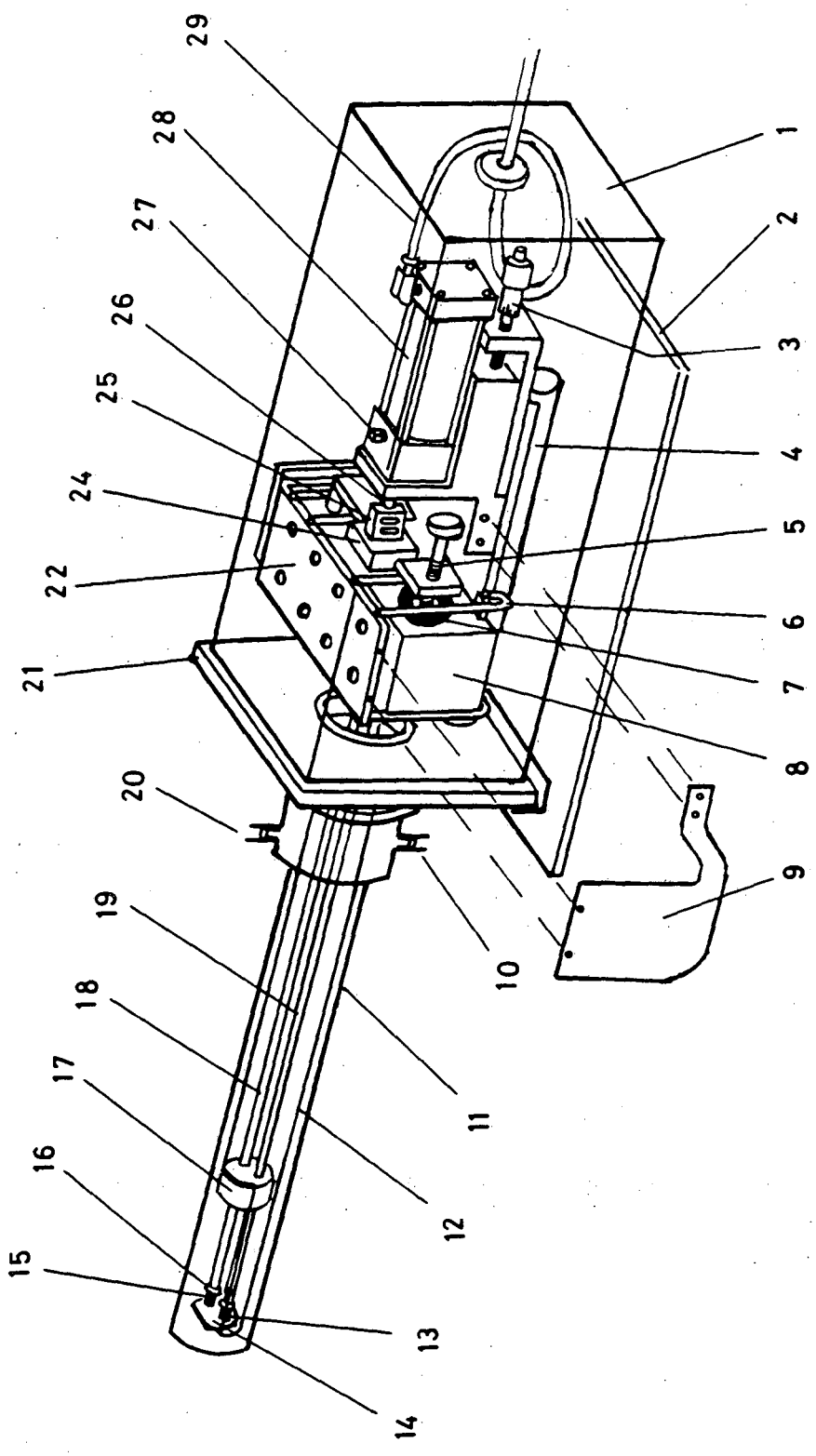

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Fig. 1(a)

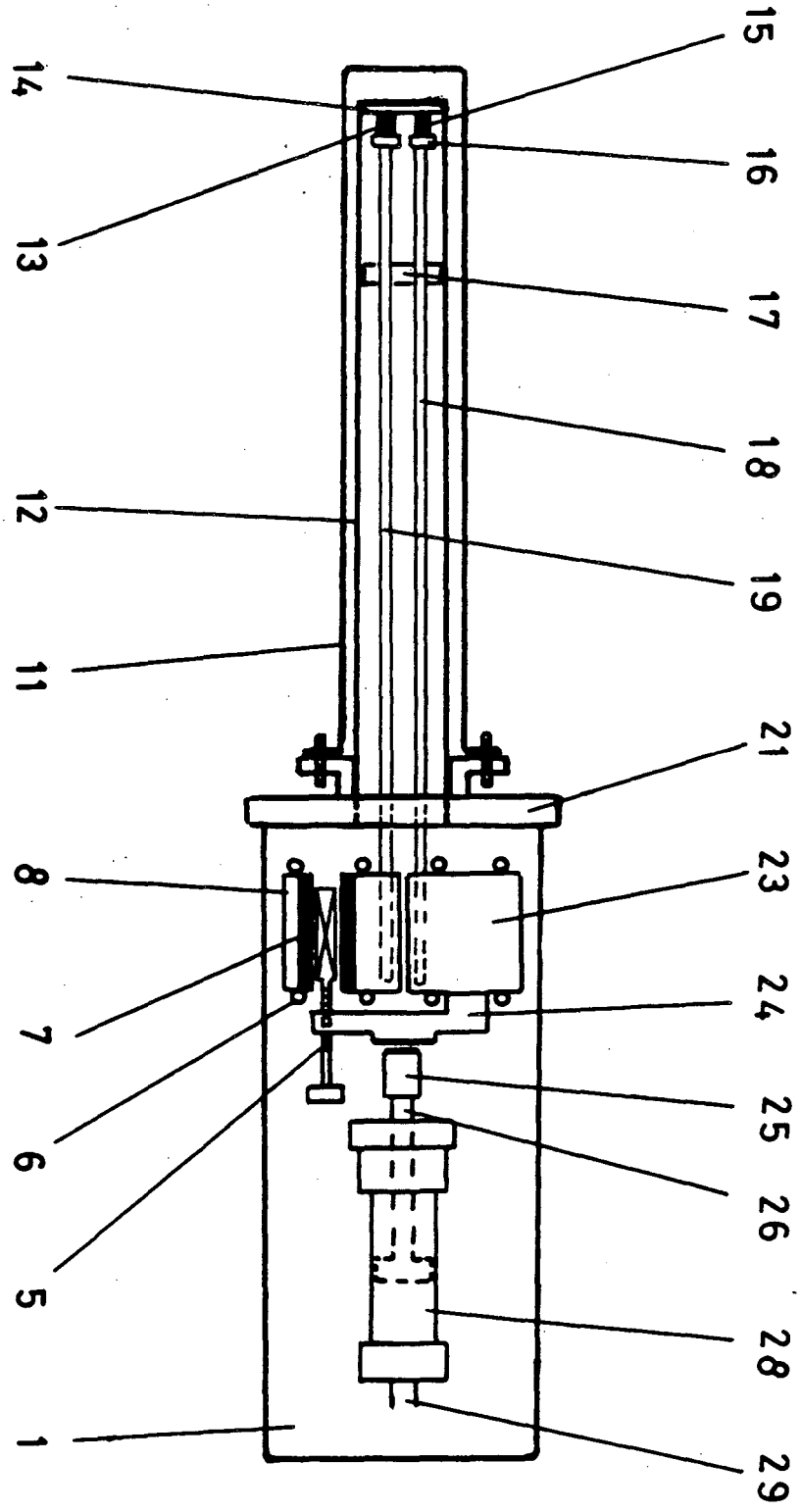

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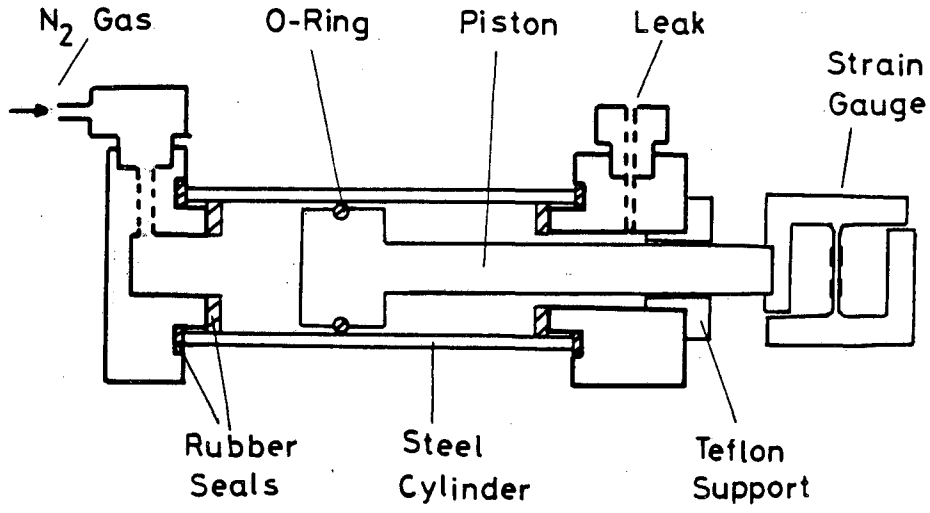
Fig. 1(b)

(a)



(b)

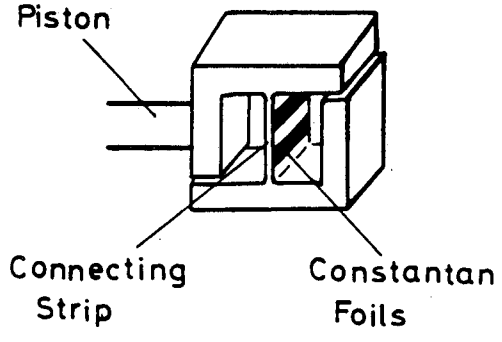


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Fig. 2(a), 2(b)

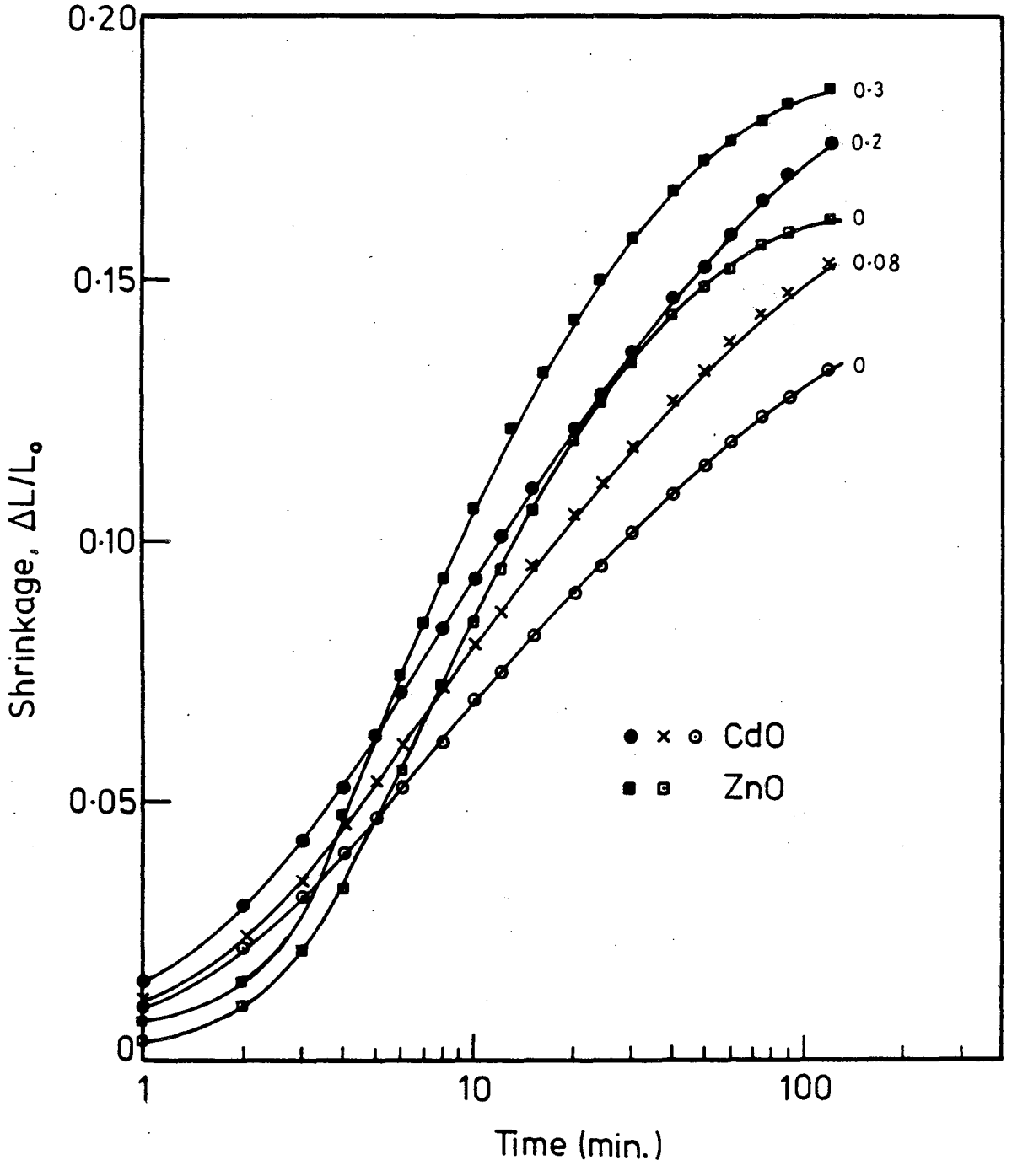

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Fig. 3

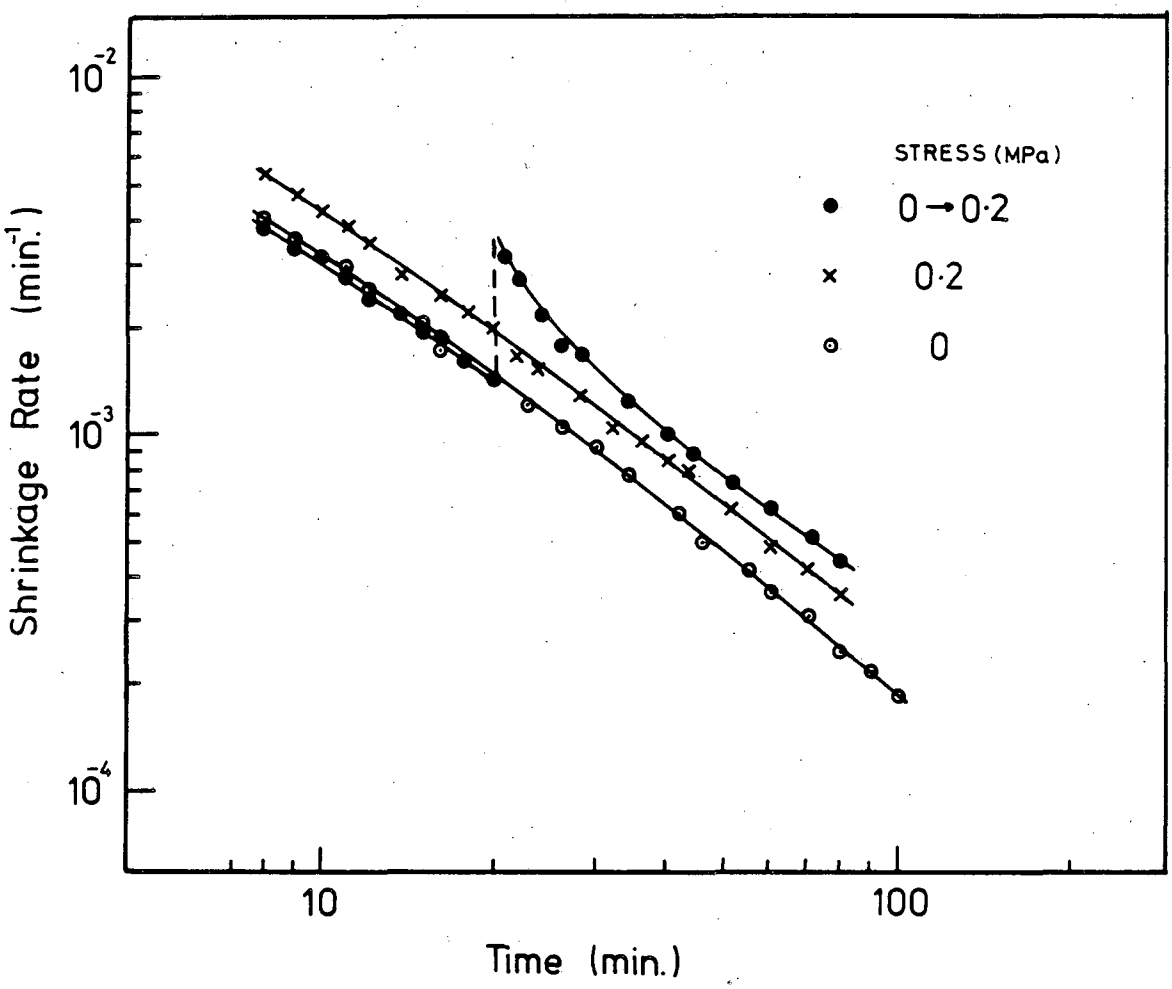

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Fig. 4

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