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Thermal Expansion Measurements on Creep Tested Mg-PSZ

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ABSTRACT

Thermal expansion measurements made on tension-creep-tested Mg-PSZ show the recovery of much of the plastic strain that was induced by the creep test. The amount of plastic strain recovered on the initial measurement was found to increase with the time between the tension-creep experiment and the initial thermal expansion measurement. Times in excess of 100 days caused the recovery of more strain in the thermal expansion measurement than had initially been induced in the tension-creep test. This aging process was accelerated by heating to 400°C. These results are in qualitative agreement with an earlier neutron diffraction measurement.

INTRODUCTION

Pure zirconia (ZrO_2) has three stable polymorphs: monoclinic (m), tetragonal (t) and cubic (c). At room temperature the m phase is stable, with the transformations m $\rightleftharpoons$ t and t $\rightleftharpoons$ c occurring at 1200°C and 2400°C respectively. The t→m transformation is martensitic (Wolten, 1963, Bansal and Heuer, 1972) with a volume expansion of ~4%. Cycling through the m $\rightleftharpoons$ t transformation places pure zirconia under severe stress on account of this volume contraction and expansion. Partially stabilized zirconia (PSZ) is produced by adding oxides such as MgO and CaO in solid solution with cubic ZrO_2. Appropriate heat treatment then develops a microstructure consisting of cubic grains and metastable tetragonal precipitates, which can transform under stress (transformation toughening). The volume expansion of the t→m transformation then leads to three- and four-fold increases in both the strength and the toughness of PSZ. Because of these desirable mechanical properties, PSZ ceramics have been the subject of considerable research over the last fifteen years (Heuer, 1987, Green et al., 1989, Rühle and Evans, 1989). The most common of the PSZs in use is MgO-stabilized PSZ (Mg-PSZ).

Plastic deformation of a sample is the permanent strain that is produced by a tensile or compressive stress and which is retained in the sample after the release of the stress. Plastic deformation measurements on Mg-PSZ are of interest since they provide information on the way that Mg-PSZ reacts to an applied stress and this will then supply information on how to improve the mechanical properties. It has been suggested that the strength of Mg-PSZ is governed by the onset of plastic deformation (Swain, 1985).

The first work reporting plastic deformation in zirconia ceramics was apparently that of Larsen in 1981 [cited in (Swain, 1985)] using a four-point-bend test and presumably measuring the plastic deformation on the tensile surface. Since this work, research has focussed on compression testing (Lankford, 1983, Chen and Reyes-Morel, 1986, Reyes-Morel and Chen, 1990, Rogers and Nemat-Nasser, 1990). There has been some other work on plastic deformation in tension tests, both in uniaxial tension (Marshall, 1986) and in four-point-bending (Swain, 1985, Marshall, 1986). All of these tests were conducted with an increasing stress. There has been little reported in the literature on testing with constant tensile or compressive stress (creep tests).

Plastic deformation occurs as a result of the volume expansion of the stress-induced t→m transformation and from the volume expansion of cracking (Chen and Reyes-Morel, 1986, Reyes-Morel and Chen, 1990). The central problem of plastic deformation measurements in Mg-PSZ is to separate the contribution to the plastic deformation from these two sources. This has been achieved for work done using compressive plastic deformation (Reyes-Morel and Chen, 1990) where it was found that cracking contributes little to the observed plastic deformation. However the same is not true for tensile plastic deformation measurements.

The contribution of the t→m transformation to the cbserved plastic deformation can be determined if the volume fraction in the bulk of the sample that transforms from t→m is measured. From knowledge of the volume expansion at the transformation this fraction is used to determine the strain accompanying transformation.

Potential methods for determination of the volume fractions of the different phases are Raman spectroscopy (Clarke and Adar, 1982), and x-ray diffraction (Garvie and Nicholson, 1972, Evans et al., 1984, Hill and Reichert, 1990) which are both techniques which measure the phase content on the surface of the sample, and neutron diffraction (Kisi et al., 1989, Howard et al., 1990) which measures the volume fractions of the phases in the bulk of the material. It has been established that on account of the metastability of the tetragonal precipitates it is not possible to produce a surface with phase content that is representative of the bulk of the sample (Hill and Reichert, 1990) thus raising doubts as to the reliability of measurements. This is due to the surface preparation process inducing transformation in the near-surface region. X-ray diffraction measurements are also made difficult in Mg-PSZ on account of overlapping peaks.

One problem with neutron diffraction has been that with the limited availability of machine time there are often long delays between creep deformation and the neutron diffraction measurement to determine the fraction of transformation. This is a problem since it was observed that a time lag between that from an initial neutron diffraction measurement and that from a similar neutron diffraction measurement eight months later, produced an increase in the amount of transformation from ~1% to ~5-6% (E.H. Kisi, personal communication, 1991).

Thermal expansion can be used to determine the amount of monoclinic zirconia since heating causes the m→t transformation and the volume decrease at the transformation is detected with thermal expansion (Dworak et al., 1986).

EXPERIMENTAL METHOD

Plastic deformation measurements were made on Mg-PSZ under conditions of constant uniform tensile stress. The details of room temperature creep experiments are reported elsewhere (Finlayson et al., 1991). There has been little reported in the literature on this area of plastic deformation measurements on Mg-PSZ. Such measurements will be referred to as tension-creep measurements. The materials tested were from three separate batches of nominally TS grade Mg-PSZ[1]. There were differences in grain size between the batches and this in turn lead to differences in plastic deformation behaviour. It was noted that the larger the average grain size the larger the plastic deformation that could be induced in the sample before failure occurred. Plastic deformation measurements in tension were made with cylindrical samples in which the gauge length, typically 40mm, had a diameter of 6mm compared to a chuck di-

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[1] supplied by ICI Advanced Ceramics, Clayton, Australia

ameter of 12mm. Plastic strains were measured during deformation, for both the longitudinal (along the stress direction) and the transverse (around the circumference of the cylinder) directions, using strain gauges. The deviation amongst the readings from separate strain gauges mounted around the circumference of the sample was usually less than 5% so that the applied tensile stress was very close to uniaxial. After fracture, the plastically deformed section was cut free using a diamond saw for thermal expansion measurements. Plastic strains at fracture varied from 0 to ~5 × 10^{-3}, depending on the batch that was tested.

Thermal expansion measurements were made using a commercial pushrod dilatometer capable of measurements up to 1500°C[2]. The data were not corrected for the expansion of the support since the measurements made were the differences between heating and cooling curves and thus the expansion of the support cancelled out. The calibration was checked using several samples of known expansions.

Thermal expansion for Mg-PSZ after plastic deformation was measured from room temperature to 700–800°C. The time between plastic deformation in tension and the initial thermal expansion measurement varied between 10 and 460 days. From these it was hoped to investigate the time dependence of the stress-induced microstructure which had been noticed in neutron diffraction, as referred to above.

RESULTS AND DISCUSSION

Figure 1 shows a thermal expansion measurement on the as-received material. There is a small hysteresis between the heating (top) and the cooling (bottom) curves on account of a small amount of monoclinic zirconia transforming to tetragonal upon heating, starting at about 500°C, and then reverting back to monoclinic upon cooling. The hysteresis is seen between the heating and the cooling curves as there is a temperature hysteresis between the forward and the reverse transformations. The temperature of the transformation is lowered from that of the monoclinic to tetragonal transformation in pure zirconia on account of the constraining effect of the surrounding cubic matrix (Hannink and Swain, 1986). The transformation appears to be fully reversible as repeated runs produced results identical to this one.

When thermal expansion measurements were made on Mg-PSZ plastically deformed in tension, it was discovered that heating through the 500°C region caused the reversal of most of the plastic strain that had been produced in the creep test. This is shown in Figure 2. The microscopic origin of this strain

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[2] Di 24 model, Adhamel Lhomargy

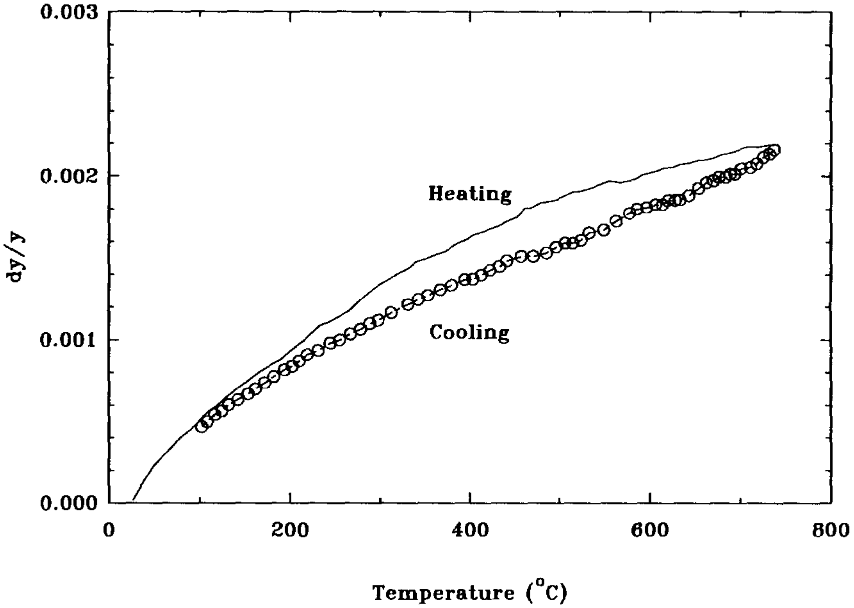

Figure 1: Thermal expansion measurement on as-received TS Mg-PSZ. The small amount of hysteresis between the heating and the cooling curves arises from the t→m transformation.

recovery is suggested to be the m→t reversion and crack closure. The m→t reversion is expected to occur at this temperature as this is the equilibrium temperature for the m→t transformation for intragranular monoclinic particles. Crack closure has been indirectly observed through Young's modulus measurements. In those measurements it was found that heating a plastically deformed sample of Mg-PSZ to 800°C caused an increase in the Young's modulus of the sample (A.K. Gross, personal communication, 1991). This is expected to arise from a reduction in the amount of cracking in the sample (Reyes-Morel and Chen, 1990).

The reversal of plastic strain seen in Figure 2 is permanent and is observed as a large, approximately constant hysteresis between the heating and the cooling curves below 400°C. A quantitative measure of this hysteresis is read directly as a strain, from the separation of the heating and cooling curves. Subsequent thermal expansion measurements on this sample then show a small amount of reversible monoclinic to tetragonal transformation and are thus similar to the thermal expansion measurement made on the as-received material (Figure 1). For this sample the measured hysteresis is 1.34×10^{-3}.

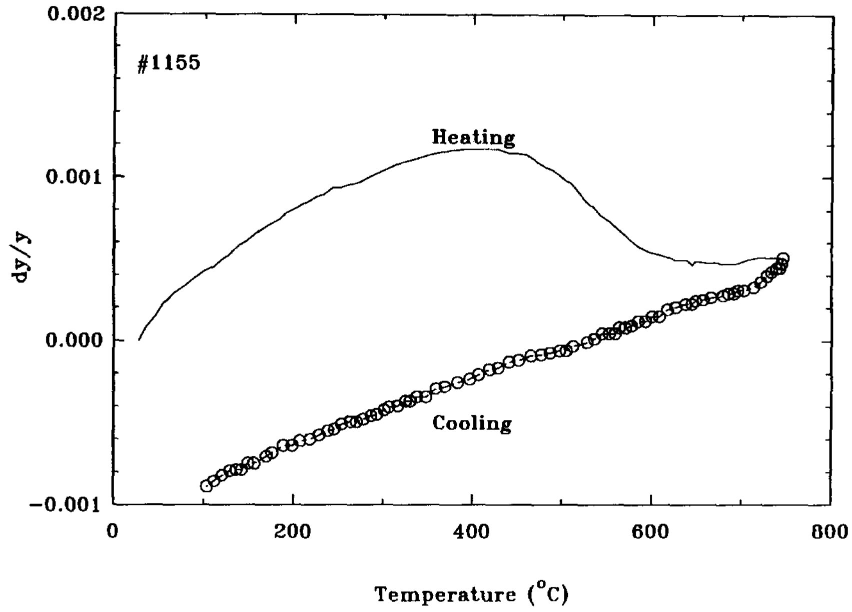

Figure 2: Initial thermal expansion measurement on tension-creep-tested Mg-PSZ. A large hysteresis is observable between heating and cooling curves.

The actual plastic strain measured along this direction during the creep test was 1.5×10^{-3}.

It is clear that almost all of the plastic strain induced by the plastic deformation test has been relieved by heating above 500°C. An effect similar to this has been observed previously in studies on plastic deformation under four-point bending. In these measurements the permanent bending produced by the four-point-bend test was eliminated by heating the sample beyond 500°C (Swain, 1988).

Thus the changes in the amount of plastic deformation can be monitored as a function of time by varying the time between the tension-creep test and the first thermal expansion measurement. In order to compare samples that had different plastic strains induced by the tension-creep experiment, the ratio of the plastic strain released by the thermal expansion test (released strain) to the plastic strain induced by the tension-creep experiment (induced strain) was calculated for each experiment. This ratio was then plotted against the time between the two tests. This result is shown in Figure 3. These results are plotted as circles in Figure 3. An examination of this plot shows that there

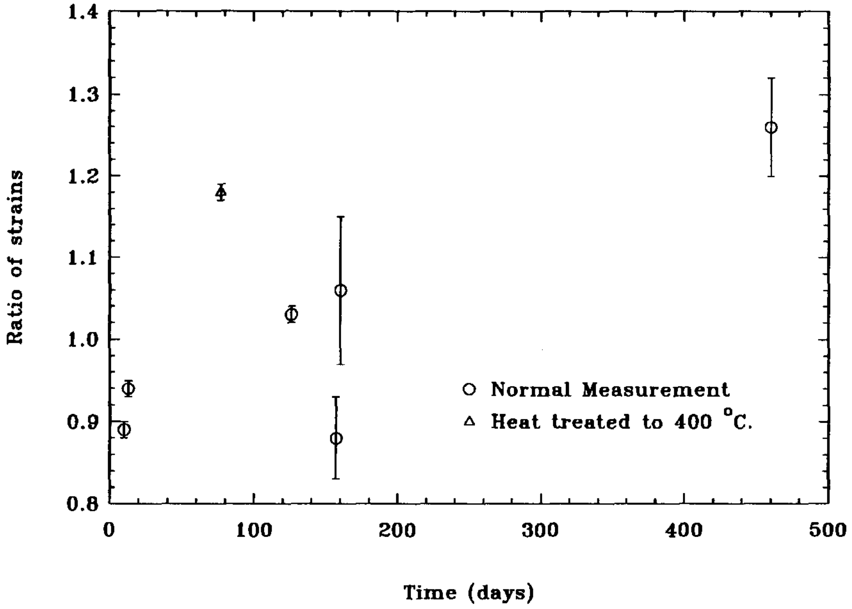

Figure 3: Ratio of released strain to induced strain as a function of the time between the plastic deformation measurement and the thermal expansion measurement.

is indeed a clear time dependence. Dilatometer measurements made shortly after the tension-creep test (~ 10 days) show a recovery of most (~90%) of the plastic strain induced by the tension-creep experiment. As the time between the tension-creep test and the thermal expansion measurement increases, this ratio also increases until the amount of strain recovered in the dilatometry measurement is larger than the plastic strain that was induced by the tension-creep experiment. The single measurement plotted with a triangle is one in which this aging process was accelerated by heating to 400°C. This time dependence is in qualitative agreement with the neutron diffraction data which showed an increase in the monoclinic content as a function of time (E.H. Kisi, personal communication, 1991).

There seems to be little doubt that the time dependence observed once in neutron diffraction is a real effect. Presumably the effect of the stress has been to destabilize the tetragonal precipitates enough so that they continue to transform after the stress has been removed. The effect cannot be a diffusional effect since diffusion is very sluggish at 1100°C and thus would be virtually non-existent at room temperature (Grain, 1967). This effect was studied

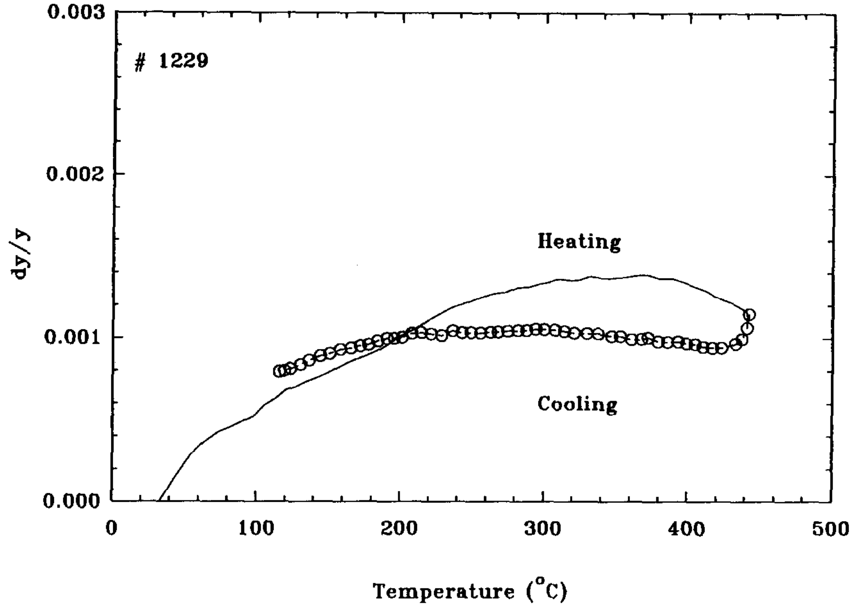

Figure 4: Initial thermal expansion measurement to 400°C. This measurement was repeated three times before the thermal expansion measurement to 800°C was made.

further by aging one sample to 400°C, which was done by recording four dilatometry measurements from room temperature to 400°C. The first of these measurements is shown in Figure 4. The time between the creep tension test and the first of these measurements was ~75 days. After these a full thermal expansion measurement to 800°C was made and the result was plotted as the triangle in Figure 3.

Figure 4 shows clearly how the thermal expansion to 400°C has accelerated the aging process that occurs at room temperature and is shown in Figure 3. The heat treatment to 400°C has produced an expansion in the length of the sample. This can be observed from where the cooling curve crosses over the heating curve. While the cooling trace was not measured to room temperature, it is clear that the measurement has caused an increase in the length of the sample. But more importantly heating the sample a further 200°C causes the reversion of all the plastic strain that was induced in the original test as well as this thermal expansion measurement.

There is thus the possibility of using these thermal expansion measurements to correct the neutron diffraction data for the time between the tension-creep experiment and the neutron diffraction measurement. However it is clear that further work to quantify this effect is needed.

CONCLUSION

After creep in tension for Mg-PSZ, the plastic strain was recovered by heating to ~600°C. The amount of recovered strain can be measured with thermal expansion. The recovered strain was found to increase with the time between the initial measurement and the first thermal expansion measurement. This was found to be a very slow process, but could be accelerated by heating to just below the reversion temperature. This effect is not well understood at present. These results are in accord with previous neutron diffraction measurements.

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