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Influence of Whisker Reinforcement on the Creep Behaviour of Zirconia Matrices

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Abstract

Zirconia matrices reinforced with 13 vol% SiC whiskers, obtained either by hot-pressing or by extrusion, have been deformed by compressive creep. The composite behaviour is similar to that of the matrix alone and suggests identical deformation mechanisms. The creep rate was not much affected by the whiskers; in particular the creep rate was not dependent on the direction of the compression axis. By contrast, for some directions of the compression axis, a strain anisotropy appeared in the plane perpendicular to the compression axis. The influence of whisker orientation on deformation has been explored by considering the matter flows in the vicinity of whiskers and the whisker distribution.

1 Introduction

This paper deals with the high temperature creep behaviour of zirconia matrices reinforced with SiC whiskers. Its main concern is with the understanding of the interactions occurring between a creeping matrix and a hard, nondeformable phase when large strains are achieved.

In particular, in a recent study concerning the superplastic deformation of an alumina–zirconia matrix reinforced with 25 vol% SiC whiskers,[1] a strong influence of the whisker orientation on the creep rate and on the shape of strained samples was observed. For instance, samples whose compression axis (CA) was perpendicular to the hot-pressing axis (HPA) showed a strain anisotropy in the plane perpendicular to the compression axis, the two transverse strains (the one parallel to the HPA over that in the perpendicular direction) being in the ratio 2.

Such results have been rarely reported in studies concerned with the deformation of ceramic matrices reinforced with SiC whiskers either because deformation was conducted in four-point bending,[2–5] a test whose analysis is complex, or, for the few studies conducted in compression, because the orientation effect was not systematically studied.[6,7] Only Lorenz[8] and Swan *et al.*[9] investigated the influence of whisker orientation on the resultant creep rates. Lorenz studied the effect of whisker orientation for two whisker contents: 20 and 29 vol%. For the first quantity no influence of the compression axis orientation was observed, while for the second, the creep rate of specimens with the CA perpendicular to the HPA was found to be about 4 times faster than the creep rate of samples parallel to the HPA in agreement with our own results.[1] Concerning Swan *et al.*'s work, no effect of sample orientation on the creep rate was observed by these authors. These various results show the complexity of the influence of whisker orientation on the resultant deformation.

From the theoretical point of view, models accounting for enhancement of creep resistance are mainly based on stress transfers across the matrix–whisker interfaces.[10–13] At a high temperature, however, diffusion can be fast enough to partially release shear stresses on the interfaces. Moreover, models predicting an influence of the whisker orientation are seldom in agreement with experimental results.

In point of fact, the above results show that the effect of whiskers on the deformation rate cannot be reduced to a simple stress transfer effect across interfaces but that secondary effects such as whisker interaction, local plasticity or direction of matter flow, must be taken into account. It is partially with this object that this work was undertaken.

In this paper the results of creep tests on a SiC_w-reinforced zirconia composite compacted by various techniques are reported. Mechanical results and microstructure observations are first

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presented. Then, the anisotropy effects are discussed, not specially for this material but more generally with reference to literature data.

2 Experimental Procedure

The material studied was a 3 mol% yttria stabilized zirconia matrix (Tosoh, Japan) reinforced with 13 vol% of both α and β SiC whiskers (Tokai Carbon, Japan). Two kinds of dense materials (ρ = 5·72 g/cm^3) were obtained at the Ecole Polytechnique Fédérale de Lausanne (Switzerland): either disks, hot-pressed at 1450 or 1550°C (to obtain various zirconia grain sizes), or cylinders extruded at 1600°C through a conical graphite die.[14] This last method partially explains why a zirconia matrix and a relatively low whisker content were used. These two techniques entail different whisker distributions. In the first case, the whiskers were almost perpendicular to the hot-pressing axis whilst in the second case, they were more or less aligned in the extrusion direction (ED).

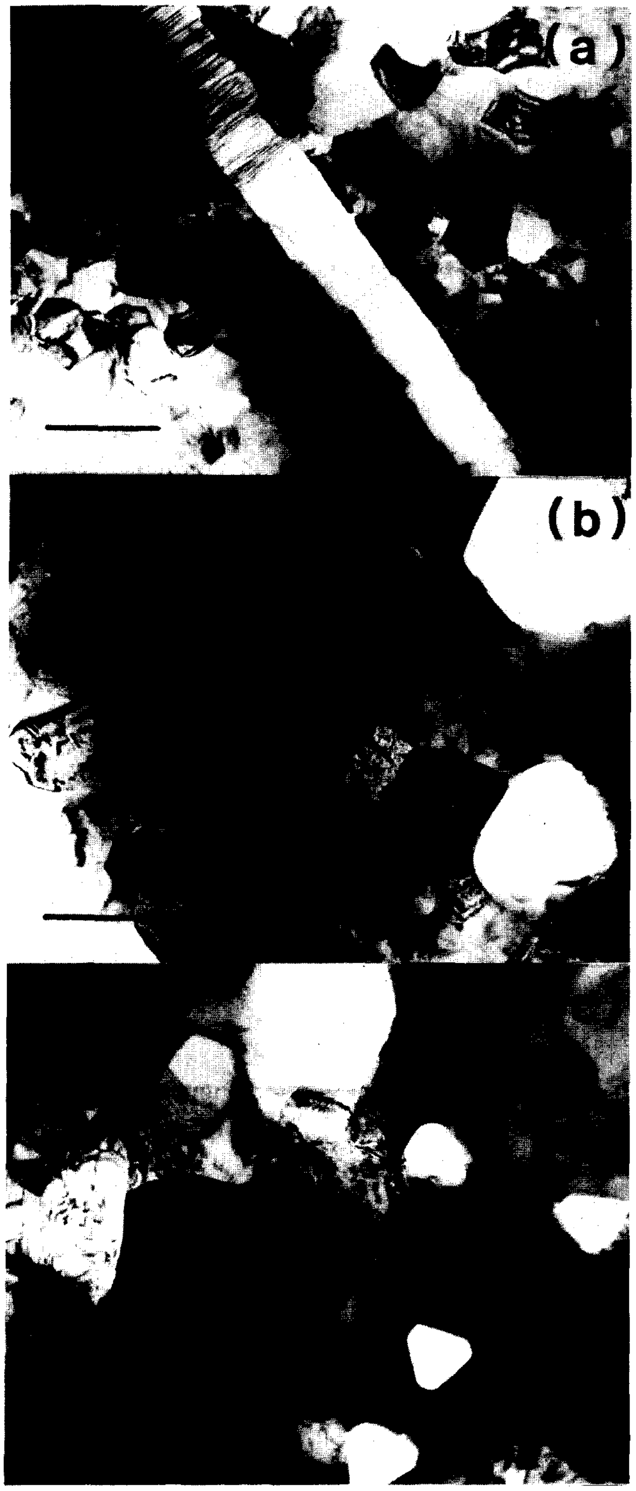

Fig. 1. Typical sample microstructures (scale bar = 0·5 μm): (a) HP at 1450°C; (b) HP at 1550°C; (c) extruded at 1600°C.

Microstructures of the three reinforced materials are shown in Fig. 1. The mean grain sizes of zirconia were respectively 0·33 and 0·54 μm in the disks hot-pressed at 1450 and 1550°C and 0·85 μm in the extruded material. The whisker diameter varied between 0·2 and 1 μm and the maximum length was 10 μm. The average aspect ratio was about 10. No glassy phase was detected by conventional transmission electron microscopy (TEM) at the matrix–whisker interfaces or in the matrix, but the presence of a thin intergranular film (~1 nm) in this kind of material is not to be excluded.

Due to the preferential whisker orientations, specimens with the compression axis either parallel or perpendicular to the hot-pressing axis or to the extrusion direction were cut, resulting in the four sample configurations of Fig. 2, called respectively P//, P⊥, E// and E⊥. These configurations correspond

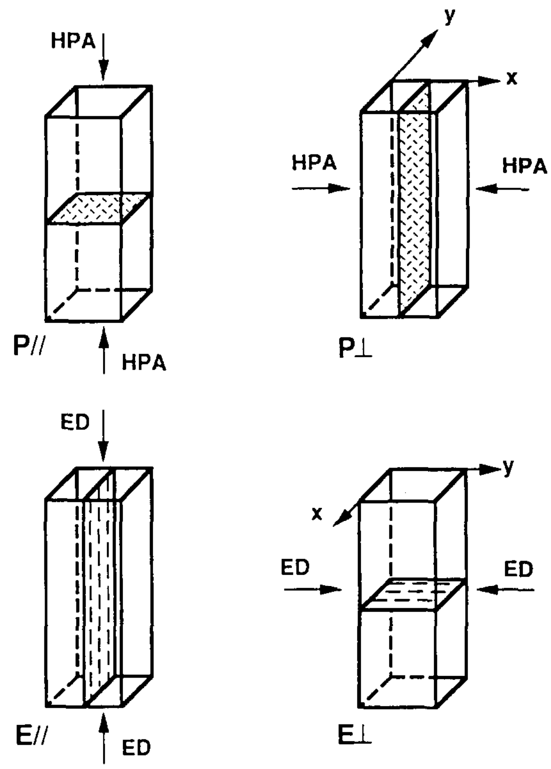

Fig. 2. Configurations of samples used in this study. P stands for hot-pressed specimen (HPA: hot-pressing axis) and E means extruded specimen (ED: extrusion direction). In each instance, the compression axis lies parallel to the long dimension of the sample.

to whiskers lying in planes perpendicular or parallel respectively to the compression axis for P samples, or to whiskers respectively parallel or perpendicular to the compression axis for E specimens. Sample size was typically 3 × 3 × 7 mm^3.

Compressive creep experiments were conducted in an argon atmosphere between 1260 and 1330°C over a stress range of 20–120 MPa. Final strains varied typically between 20 and 40%. Two or even three specimens were tested for each deformation condition.

After creep tests, microstructure observations were made by TEM at 200 kV or SEM (scanning electron microscopy) at 15 kV on polished and thermal etched surfaces.

3 Experimental Results

3.1 Macroscopic behaviour

At first, to test the effect of whisker orientation on the creep rate, experiments were conducted on the two kinds of materials (hot-pressed or extruded) at several temperatures in the same stress conditions. Figure 3(a) is a plot of creep rate versus true strain for two specimens, a P// and a P⊥, deformed at 1300°C and 40 MPa. The two curves are very similar. The same inference can be drawn for E specimens as presented in Fig. 3(b) for two tests conducted at 1330°C and 30 MPa. These results do not show a significant influence of the compression axis orientation relative to the whisker direction on the creep rate. Under these conditions, the different specimen configurations can be neglected and we will assume later on, in particular in the creep rate analysis, that various specimens differed only by their grain sizes.

By contrast, in the case of P⊥ and E⊥ samples, transverse strains were whisker orientation dependent. As previously observed,[1] for these two kinds of specimens the transverse strain in the x direction in Fig. 2, the direction perpendicular to the whiskers, was systematically larger than that in the other transverse direction (y direction). The corresponding strain ratio, ϵ_x/ϵ_y, was approximately 1·5 whatever the deformation conditions. *A priori*, the insensitivity of creep rates to whisker orientation may seem to be inconsistent with the above strain anisotropy. This will be discussed later.

The experimental steady-state creep rates $\dot{\epsilon}$ were analysed according to the phenomenological relationship:

$$\dot{\epsilon} = A\ (\sigma^n/d^p)\ \exp\ (-Q/RT) \qquad (1)$$

where σ is the stress, d is the zirconia grain size, n and p are the stress and grain size exponents respectively, Q is the creep activation energy, R and T having their usual meaning. A is a constant depending on the microstructure and on the controlling rate mechanism.

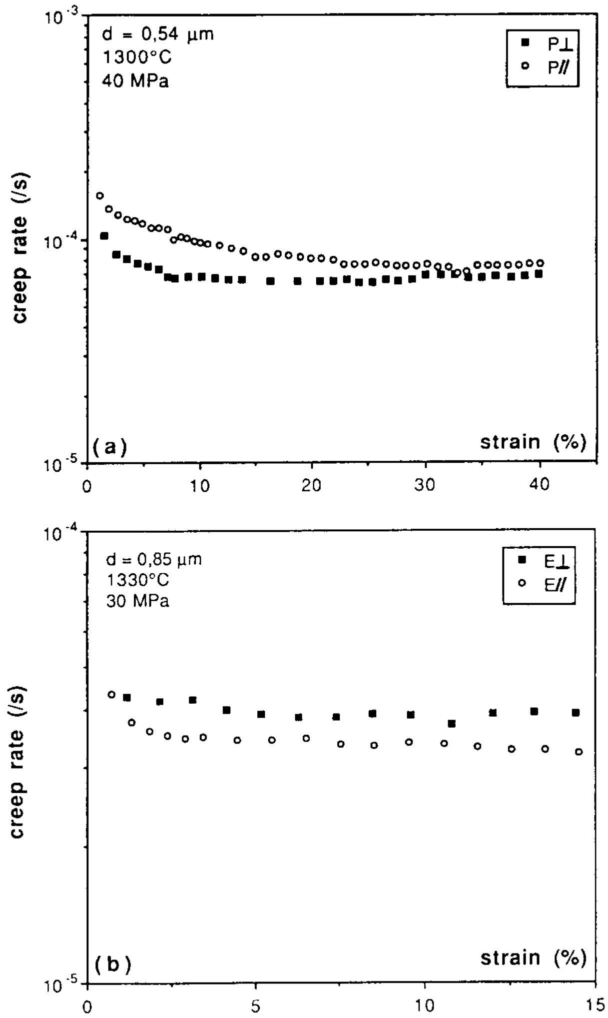

Fig. 3. Influence of whisker orientation on the creep rate: (a) case of two hot-pressed specimens deformed at 1300°C, one parallel and one perpendicular to the hot-pressing direction; (b) case of two extruded specimens deformed at 1330°C, one parallel and one perpendicular to the extrusion direction.

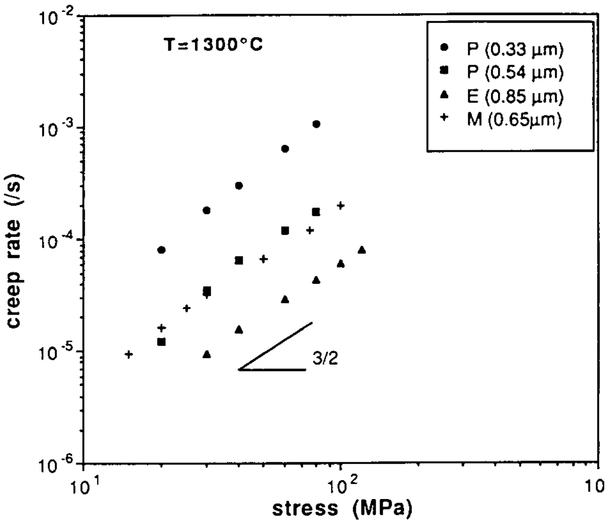

Fig. 4. Influence of stress on the creep rate at 1300°C of the three reinforced materials (P and E specimens). The curve with the open points represents the matrix behaviour (grain size = 0·65 μm).

The effect of the stress on the creep rate is presented in Fig. 4 as a log–log plot of the steady state creep rate versus stress for the three tested grain sizes. On this diagram, the experimental points concerning the zirconia matrix[15] (grain size 0·65 μm) have been added. A great similarity with the present experimental curves is observed. For the high stress range (typically higher than 40 MPa), the stress exponent has a value of 1·6. At the lowest stresses, especially for the two kinds of hot-pressed samples which have finer grain sizes than the extruded material, the stress exponent n slightly increases, as for the matrix.[15] However, the difficulties associated with conducting creep tests at very low stresses (5–10 MPa) do not allow us to investigate this stress range and to study the possibility of an increasing stress exponent in this domain or the occurrence of a threshold stress such as has been observed in creep studies of alumina–zirconia matrices reinforced with 27 vol% of SiC whiskers.[1,16,17]

From Fig. 4, the effect of the grain size on the creep rate was determined over the range 30–80 MPa (Fig. 5). The grain size exponent has a value between 3 and 4, being relatively constant throughout the entire stress range.

The temperature dependence has been analysed from temperature changes performed during tests at a constant stress of 30 MPa. The resultant activation energy does not depend on the specimen kind and ranges between 550 and 600 kJ/mol, the same activation energy as found for the matrix in the high stress regime.

3.2 Structure development

TEM and SEM examinations showed that the microstructure of crept samples was nearly identical to the microstructure of undeformed specimens. No glassy phase related to whisker oxidization

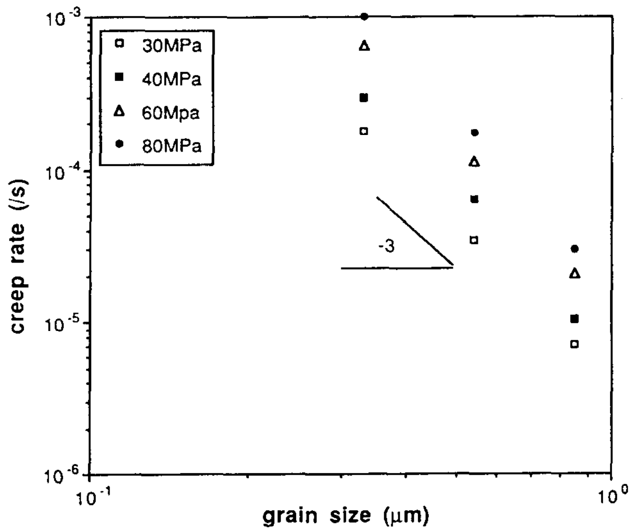

Fig. 5. Influence of grain size on the creep rate at 1300°C of reinforced materials.

Fig. 6. Microstructure inside the zirconia matrix after a strain of 40% at 1300°C for a specimen pressed at 1550°C. Scale bar = 0·5 μm.

during the deformation test, was detected at the whisker–zirconia interfaces or in the matrix by conventional TEM. SiC whiskers presented no evidence of plastic deformation such as shape change or intragranular dislocation activity. Zirconia grains which retained their initial equiaxed shape and size were generally dislocation free (Fig. 6). On the other hand, boundary dislocations were observed, especially in the extruded material. The main structure changes concerned the whisker orientation relative to the compression axis and the cavity nucleation.

During deformation of P⊥ and E// specimens, for which whiskers do not lie in planes perpendicular to the compression axis at the test onset, the angle between compression axis and mean whisker direction increased with strain. Thus, at a strain of 40%, more than 70% of the whiskers were about perpendicular to the compression axis, in good agreement with our previous results.[1] Moreover, in the case of P⊥ specimens, whiskers tended to stay in the planes perpendicular to the initial hot-pressing axis, rotation of whisker occurring more or less isotropically in the E// specimens. This means that during deformation the whisker distribution in P⊥ specimens became identical to that of E⊥ samples while E// specimens tended to be similar to P// samples.

Cavity formation was observed at every stress with an increasing tendency at high stress. The cavitational component of creep strain was determined by density measurements. According to the deformation conditions, the observed decrease in density ranged from 0·9 to 3·6% as shown in Table 1. Cavities were mainly observed at the matrix–whisker interfaces in the matrix phase (Fig. 7) but sometimes also inside the zirconia phase itself. They were generally located at triple points but could also be developed along boundaries. In this case, porosity was preferentially oriented parallel to the stress direction. When a creep acceleration was observed, it was correlated to pore coalescence and microcrack formation (Fig. 8).

Table 1. Decrease in density $\Delta\rho/\rho o$ at 1300°C as a function of experimental conditions. P* means hot-pressed at 1550°C

Specimen	*Stress (MPa)*	*Creep strain*	$\Delta\rho/\rho o$ (%)
P//	20–30–40	0·41	1·9
P//	30	0·36	2·1
P//	40–60–80	0·36	3·1
P⊥	20–30–40	0·43	1·6
P⊥	40	0·40	2
P⊥	40–60–80	0·36	3
P*//	20–30–40	0·34	1·8
P*//	40–60–80	0·34	3·2
P*⊥	40	0·40	0·9
E//	30	0·33	1·3
E//	30–40–60	0·26	2·4
E⊥	30–40–60	0·19	1·9
E⊥	40–80–120	0·29	3·6

Fig. 7. Crack at a matrix–whisker interface in an extruded sample deformed at 1330°C up to a strain of 34%. The arrow is parallel to the compression axis. Scale bar = 0·5 μm.

Fig. 8. Observation of microcrack coalescence during the tertiary creep of a specimen deformed at 1300°C under a stress of 120 MPa. Scale bar = 1 μm.

4 Discussion

4.1 Creep mechanism

As shown in Fig. 4, the stress influence on the creep rate of the three reinforced materials is nearly identical to that of the zirconia matrix. The creep rate is characterized by a stress exponent whose mean value of $n \sim 1{\cdot}6$ does not reflect a single mechanism, but rather a combination of two or more active mechanisms.

TEM examinations and density measurements showed that the contributions of intragranular dislocations and cavitation to creep strain was likely to be too weak to account for such a stress exponent. In addition, any tendency to an increase in the stress exponent should be observed at high stress and not at low stress, if cavitation and/or dislocation contributions were significant.

The activation energy, 550–600 kJ/mol, is similar to that of the matrix in the high stress range;[15] this suggests:

(1) That whiskers do not have a significant influence on the controlling mechanism of creep, in agreement with the fact that the creep behaviour was only little affected by the whiskers.
(2) That only mechanical interactions between matrix and whiskers and not chemical interactions (with consequent changes in the diffusion mechanisms and activation energy) took place during deformation.

The grain size exponent is also coherent with the value obtained in the high stress range for the matrix.

Finally, except for the A factor in eqn (1), all the thermomechanical parameters of the composites, n, p and Q, are approximately the same as for the matrix. The deformation mechanisms are therefore believed to be identical for the reinforced and unreinforced materials: grain boundary sliding and a grain rearrangement process are responsible for a large amount of deformation. If the specific nature of the accommodation mechanisms cannot be unambiguously established on the basis of the exponents and activation energy observed and owing to the lack of reliable diffusion data, some informations can be obtained from literature:

(i) a comparison of our creep rates with those expected from Nabarro–Herring diffusion creep[18] shows that ours are faster by more than two orders of magnitude suggesting in agreement with Chokshi's conclusion[19] that N–H diffusion creep should not contribute significantly to deformation of our materials;
(ii) in addition, according to several authors[20–22] the grain size of our specimens lies in the range where grain boundary diffusion should be expected.

Nevertheless, the effect of impurities and the possible presence of a thin intergranular film must not be neglected in the deformation accommodation.

With respect to the matrix behaviour,[15] whiskers behaved as an inert and non-plastic phase; their effect appears mainly: (i) in the difference of transverse strains for P⊥ and E⊥ samples and (ii) in the creep damage. For the first point, this means that the whiskers reduce the deformation in the direction parallel to the whisker axis. This point is more specially discussed in the next part. Concerning the second point, this confirms the absence of plasticity of the whiskers at the test temperatures resulting from the low diffusion coefficients and from stress levels insufficient to activate dislocation glide in the SiC phase.[23,24] Under these conditions, stress concentrations in the vicinity of the whiskers can be released only by plastic deformation in the zirconia phase. When zirconia plasticity is not efficient enough, cavity nucleation occurs.

4.2 Effect of whisker orientation on the creep rate

In contrast to our previous results,[1] the creep rates obtained in the present study were not significantly affected by the orientation of the compression axis relative to the whisker direction.

Nevertheless, in these two works, a similarity has been noted that concerns the strain anisotropy in the planes perpendicular to the compression axis in P⊥ and E⊥ specimens. (Recall that in these specimens, the whiskers were, or became during deformation, parallel lying in planes perpendicular to stress). The ratio between the two transverse strains, the strain in a direction perpendicular to the whiskers (ϵ_x in Fig. 2) over the strain in a parallel direction (ϵ_y), is dependent on the whisker content, 1·5 presently versus 2 previously.[1] This anisotropy results from a greater ease of grain rearrangement perpendicular to the whiskers, due to an easier rotation of whiskers around their own axis than around a perpendicular axis.[25] The effect of the whisker content can be understood if we consider that the influence of a given whisker on the matrix deformation decreases with the distance from the whisker. Consequently at a distance r_o of a few grain sizes, one can assume that the whisker has no more effect on the matrix behaviour and deformation becomes again isotropic in a plane perpendicular to the compression axis. That influence occurs in a cylindrical volume of radius r_o and length approximately equal to the whisker length, called interaction volume in what follows. In this interaction volume, the rearrangement rate in a direction parallel to the whisker axis slowed down more and more with respect to the perpendicular rate as the distance from the whisker decreases. At a low whisker content, two neighbouring interaction volumes do not overlap; as a result, the composite behaviour is little modified by the presence of the whiskers and the ratio between the two transverse strains, ϵ_x/ϵ_y, is roughly equal to one. But, as the whisker content increases, the overlapping, or interaction, between these volumes increases and the matrix deformation is more and more disturbed in the direction parallel to the whiskers relative to that in a perpendicular direction. Under these conditions, the strain anisotropy of P⊥ and E⊥ specimens must effectively increase with the whisker content.

Concerning the effect of whisker orientation on the sample strain rate, it can be partly understood by some earlier considerations.[1] The achievement of large strains results from matter flows whose direction is controlled by the applied stresses. As previously mentioned, in the vicinity of the whiskers, i.e. in the interaction volumes, the flows can be separated into two elementary flows (Fig. 9), one parallel to the whisker and the other one perpendicular, corresponding respectively to average contributions to the macroscopic strain rate R// and R⊥, with R⊥ > R// and R⊥ / R// increasing with the whisker content. According to the whisker content and distribution, the need to maintain the strain compatibility throughout the specimen imposes that the flows around a whisker are more or less coupled. This results also from the fact that the deformation in a layer perpendicular to the stress direction is dependent on deformation occurring in the layers just above and below due to the rearrangement or intercalation mechanism that increases the grain number in a cross sectional area and requires grain exchanges between neighbouring layers.

When all the whiskers are parallel, the same kinds of flow, those parallel or those perpendicular to the whiskers, occur around each whisker in the whole specimen. Consequently, the two matter flows can be treated independently and the resultant

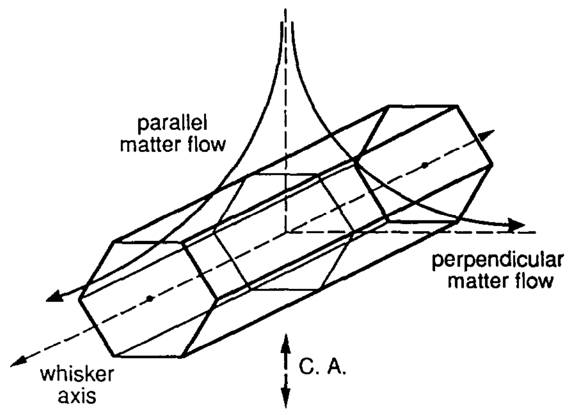

Fig. 9. Definition of parallel and perpendicular matter flows around a whisker.

strain rate should be simply the sum of R// and R⊥.

When the whisker orientations are more or less homogeneously distributed in the planes perpendicular to the compression axis, the two flows can be no more treated separately. The two extreme cases correspond to:

(i) the two flows act in parallel but the fastest one (R⊥) is slowed down by the slowest one (R//) and the resultant creep rate is estimated to twice the slowest rate. This occurs at a whisker content low;

(ii) the two flows act in series and the creep rate in them half the slowest contribution. This must correspond to high whisker contents.

Such assumptions can account, at least qualitatively, for our results at low and high whisker contents. However the above estimations assume that after hot-pressing all the whiskers laid perpendicular to the hot-pressing direction. When it is not the case, for instance when whiskers with a high aspect ratio, for which the interference threshold is reached at a relatively low content,[26] are used, the deformation of specimens with the compression axis perpendicular to the hot-pressing direction is then dependent on the whiskers out of planes perpendicular to the hot-pressing axis. Consequently in such specimens, the behaviour anisotropy related to the compression axis orientation should lessen or even disappear. The influence of whiskers on the creep rate is then strongly dependent on their orientation and morphology and cannot be simply related to their content.

5 Conclusion

The deformation of zirconia matrices reinforced with 13 vol% of SiC whiskers has shown that the creep behaviour was not strongly affected by the presence of the whiskers. The activation energy, the stress exponent and the grain size exponent were found to be similar to those of the matrix, suggesting that the same microscopic mechanisms were rate controlling. Cavity formation made a limited contribution to the deformation (a few percent for strains of 40%).

The influence of whiskers on deformation was particularly observed on the strain anisotropy of samples where the compression axis was perpendicular to the directions of hot-pressing or extrusion. The strain in a direction parallel to the whisker axis was systematically lower than that in a perpendicular direction. This observation was analysed by a greater ease of grain rearrangement when it occurs in a direction perpendicular to the whiskers. By comparison with our previous results,[1] it appeared that an increase in whisker content increased the interaction between the matrix and the whiskers and had a more pronounced effect on deformation in the direction parallel to the whiskers than in the perpendicular direction: the strain anisotropy was then dependent on the whisker content for these kinds of specimens.

The influence of specimen orientation on the creep rate has been explored by estimating the more or less high connection between the two elementary matter flows around the whiskers as a function of the whisker content.

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