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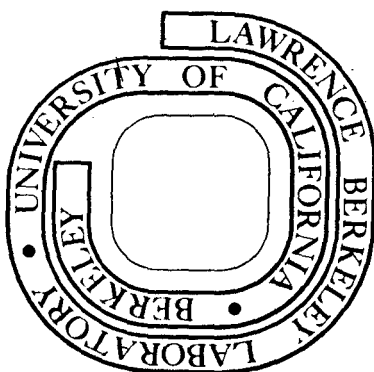
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Comment on

"Study of the Precipitation in $\text{MgO} \cdot 3.5 \text{Al}_2\text{O}_3$ During Creep Experiments"

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RÉSUMÉ

On discute des arguments développés par Duclos pour analyser la précipitation sous contrainte dans un spinelle non stœchiométrique. On montre qu'ils ne peuvent permettre de prouver si un processus non conservatif, un cisaillement périodique ou un possible troisième mécanisme est responsable ou non des caractéristiques de la précipitation d'alumine.

ABSTRACT

The arguments developed by Duclos in his analysis of stress assisted precipitation in nonstoichiometric spinel are shown to be inappropriate to decide whether glide elements, climb strain or a possible third mechanism is responsible or not for the observed features of the resolution of alumina.

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The precipitation of a rhombohedral phase (i.e. hematite, corundum,...) clearly involves two distinct concurrent processes: a geometric transformation of the anion sublattice (face centered cubic $\rightarrow$ hexagonal) and a reorganization of the cation sublattice through a diffusional event. In a recent note [1], Duclos restricted his analysis to the geometrical transformation of the anion sublattice and attempted to demonstrate that a description in terms of glide elements [2,3] could not apply to account for the related transformation. From his experimental results he concluded that the observed strain could be exclusively explained in terms of the volume contraction associated with the differences in lattice parameters between the two phases [3], and he therefore suggested an unformulated "climb strain hypothesis" to account for these results. In this short note a few comments will be made on the methodology developed in [1] as well as on the way the experimental data, derived from precipitation in $\text{MgO}(\text{Al}_2\text{O}_3)_{3.5}$ under an applied stress, have been analyzed.

Firstly, let us review the experimental procedure. Indeed, using the three different compression axes, i.e. [100], [110] and [111] does lead to substantial improvements of the previous work on nickel ferrite. However, it must be mentioned that the specimens seem to have been loaded once the temperature of the device was stable. As the creep device used in this work [4] appears to be similar to most of the others which are devoted to compression (creep or constant strain rates) of oxide materials [2,5,6], the time required for heating the specimens to the temperatures of the tests must have been of the order of a few hours (this includes the hour required for the bulk rods to reach perfect equilibrium to

ensure sufficient precision to measure unambiguously strain rates as low as 10^{-7} s^{-1}). Contrary to what happens in open systems as nickel ferrite where the beginning of precipitation is easily controlled through the partial pressure of oxygen (P_{O_2}), it must therefore be concluded that depending on the (TTT) diagram, a noticeable, uncontrolled preliminary precipitation of corundum must have occurred during the preannealing time. Surprisingly disregarded in [1], this basic aspect may lead to several objections to Duclos' conclusions, amongst which we shall briefly discuss the most serious:

- (i) the precipitation substructure shown in Fig. 5[1] could be irrelevant, and, because of the very restricted area shown here, it is possible that observing a precipitation involving these systems associated with zero glide systems could be ascribed to a precipitation artefact.
- (ii) The description of the creep curve is not realistic since a precipitation substructure has already developed and whatever its extension, it has been clearly demonstrated that it acts as a large obstacle to the development of the stress induced precipitates [2,3]; in fact, it would have been valuable to show in Fig. 4 [1] the precipitation substructure at the inflection point for a [111] compression axis instead of a [001] one, together with a control specimen submitted to an identical thermal history but unstressed.

(iii) Together with the observation of the substructure, the arguments developed in [1] are based on measurements of the strain, supposedly associated with the stress assisted precipitation. It is not obvious that the initial inflection point (Figs. 2 and 3 in [1]) of the creep curves should be associated with the early stage of precipitation, in addition we notice a serious discrepancy between Fig. 3 and the text, since it is claimed that the final stage of precipitation is associated with strain rates as low as 10^{-8} s^{-1} . Due to the uncertainties associated with the initial and final parts of this curve, the actual precipitation strain estimated from taking the derivative of Fig. 2 to transform it into Fig. 3 and then from integrating a restricted domain in Fig. 3 by extrapolation of the initial part of the curve is therefore a very rough value which is probably underestimated. Such an analysis also implies that a concurrent deformation mode (individual dislocations) occurs at 1200°C and relatively low stresses to account for the nonhatched part of the curve, a fact which shows serious disagreements with previous plastic deformation data [7,8,9]. Together with the previous objections concerning the definition of the actual extremities of the curve and its analysis, any estimation of the actual precipitation strain should take into account the fact that the total amount (ratio k in [1]) of corundum in the sample has not precipitated during the creep experiments; therefore, the parameter k' ($< k$) corresponding to the amount of stress assisted precipitates should be

determined experimentally and used instead of k in the relevant formulae. Thus we conclude that the actual strains associated with precipitation could be at least twice the values derived by Duclos. The discrepancies between his climb strain estimates and his experimental result should therefore be much larger than 80%, 21% and 3% corresponding to [001], [110] and [111] compression axes respectively.

Regarding the latter point it is believed that formula (3) in [1] should read:

$$\epsilon = k' \frac{\Delta e}{\epsilon_{sp}} \cos \alpha$$

In fact, in addition with the problem resulting from the ratio k' , there is no reason for introducing $\cos^2 \alpha$ (analogous to the Schmid factor for climb) in this very simple geometric problem. This would adequately explain why the above discrepancy is not constant with α .

Finally, an activation energy for the precipitation of corundum has been determined by temperature jumps during the creep experiments and its value is discussed in terms of ion diffusion consistent with the climb rate hypothesis. Such an analysis is basically irrelevant since it is supposed that in the classical formula employed for describing a thermally activated process (here $\dot{\epsilon} = \dot{\epsilon}_0 \exp - \frac{U}{kT}$), the pre-exponential factor remains constant during the experiments. This condition is obviously not fulfilled in this case since $\dot{\epsilon}_0$ contains a term strongly dependent upon the substructure [2,3]. This term actually varies very rapidly especially near the maximum creep rate where the measurements have been carried out [1]. Indeed contrary to what happens in steady

state creep it cannot be argued here that the rate of production of defects is equal to the rate of their elimination, since the system evolves toward complete achievement of the precipitation.

We therefore conclude that this work is too approximate to enable one to decide whether this phase transformation is achieved through glide elements or not. However, the occurrence of precipitation in a plane perpendicular to the compression axis is an unexpected property which, if it were confirmed, should yield the possibility of distinguishing between the shear and the volume contraction contributions as discussed in [3]. It would therefore allow to demonstrate that the transformation of the anion sublattice from fcc to hcp could occur through a shuffle mechanism rather than through a periodical shear as expected from the analogy with multiple phase transformations.

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