

## Grain boundary sliding in chromia thermally grown oxide



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### ABSTRACT

Residual macrostresses in chromia thermal oxide films which develop on a Ni–30Cr alloy have been investigated at room temperature thanks to Raman spectroscopy and X-ray diffraction. Combined with atomic force microscopy on the oxidised surface which gives morphological features at the chromia grain scale, stress release processes in the ceramic films are studied. It is evidenced that grain boundary sliding is activated as a companion mechanism of diffusion-creep. Such processes are sensitive to the oxide microstructure and the cooling conditions.

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Chromia or alumina forming alloys are suitable materials for high temperature applications because of their high temperature resistance which is due to the formation of slow growing and well adherent chromia or alumina surface scales respectively. However, the material durability depends on the ceramic film integrity and it is well known that these protective oxide scales are subjected to high compressive stresses [1,2]. Indeed, if the isothermal oxidation takes place within the growing scales through counter current oxygen and metal diffusion, large compressive stresses may develop in the scales. Moreover, such growth stresses combine with thermal stresses that appear during cooling step. As a consequence, the accumulation of elastic energy in the scale volume induces stress release/relief processes which may be either destructive (cracking, spallation), or not (creep of the oxide scale or the metal) [3]. The latter solution should be favoured for keeping oxide scale integrity and then its protective function. Recent studies [4–8] have shown that the microstructure in the oxide scale affects the growth stress magnitude and the relaxation mechanisms. For stressed ceramic films at sufficiently high temperatures, oxide creep is expected to occur for fine grained oxide scale rather than delamination. However, only few measurements have been reported for thermally grown oxides, in particular for chromia films while creep has been extensively studied in bulk ceramics. Measuring the stress magnitude and distribution in chromia is important because it is the primary oxide encountered in widely used stainless steels. NiCr alloy is also an oxidation resistant-chromia forming alloy

whose oxidation behaviour has been extensively studied [9–12] owing to its attractivity for fundamental investigation since the only oxide formed is chromia. Considering previous work on this alloy [13], diffusion-creep has already been indirectly evidenced in these types of chromia formers. Indeed, from the modelling of the growth stress evolution, an optimisation procedure has been used to extract the characteristic thermomechanical parameters, of the system at high temperature. In particular, it has been established that the Norton exponent for these ceramic films are close to unity which is compatible with either inter or intra granular diffusion-creep. Furthermore, the creep coefficients in the metal are systematically two orders of magnitude lower than the oxide film ones which mean that creep is occurring preferentially in the later, the film volume (proportional to the thickness) being at least one hundred times smaller in this system than the one of the metallic substrate. The development of high stresses within the oxide layer or at the oxide/metal interface will be reduced leading then to a beneficial influence on the process of scale failure. Let us notice that no feature of conventional plasticity has been evidenced in these ceramic films. The behaviour of bulk ceramics [14–17] or metals [18] during conventional creep experiments performed for specific strain–stress ranges and microstructural states, is well documented. When such a process takes place in a polycrystalline and homogeneous material, each grain is deformed. Since the material has to remain dense and coherent, the grains must move relatively to each other by grain boundary sliding. Thus, the overall deformation may be regarded as a combination of diffusion-creep and grain boundary sliding. The later effect induces specific surface topologies which have been already evidenced.

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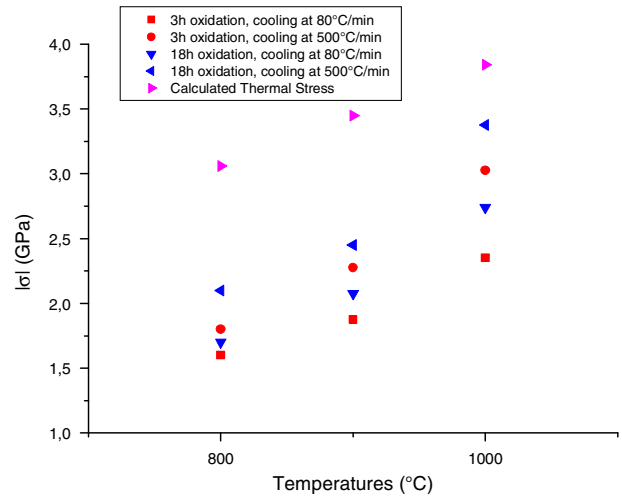
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Indeed, grain boundary sliding investigations in bulk ceramics were carried out measuring vertical offsets thanks to AFM images as reported in different studies, and also in metals in the case of superplasticity [18–22].

Considering such creep behaviour in bulk materials and the previous results suggesting diffusion-creep in chromia formers, the occurrence of concomitant grain boundary sliding in the thermally grown oxides may be evidenced leading then to the corresponding microscopic strain. Indeed, the possibility of such behaviour has already been mentioned in the literature: Kofstad [23] has explained that diffusion-creep and grain boundary sliding may occur in thermally grown oxides under compressive stress. Moon [24] and Delaunay [25] also suggest the possibility of stress release by creep and grain boundary sliding in chromia scales. And more recently Hou [26], from stress evolution measurements by XRD with Synchrotron facility, has mentioned stress release mechanisms by oxide creep or some degree of grain boundary sliding for alumina scales.

In the present work, residual stresses are determined in chromia thin films thanks to both X-ray diffraction (XRD) and Raman spectroscopy, and are combined to atomic force microscopy (AFM) measurements on the oxidised surface, in order to analyse the stress build up and release mechanisms in the scale. Following previous studies in bulk materials, surface features are investigated. Strain determination procedure is adapted to the specific case of a thermally grown oxide in mechanical interaction with a metallic substrate. The influence of the oxidation conditions, e.g. temperature and duration, and the cooling rates, on the diffusion-creep behaviour will be investigated.

1.5 mm thick disks cut in a Ni–30Cr rod have been prepared. The chemical composition of this alloy has already been given in previous work [13]. A thermal pre-treatment at 1000 °C during 1 h has been achieved to homogenise the metal. Sample surface is mechanically polished using SiC papers down to 4000 grade, then washed in demineralised water and finally dried. Oxidations have been done at 800, 900 and 1000 °C inside a muffle furnace, for 3 or 18 h. The specimens are then cooled by using different ways, the associated cooling rates being in the range [80–500 °C/min]. This later step constitutes the thermal cycling loading applied to the oxidised sample. Ex-situ stress determination has been undertaken either by XRD or Raman spectroscopy. The corresponding methodology is already described elsewhere [27]. Thanks to the good agreement between these two methods, an average stress value is given. The residual stress magnitudes in chromia films are reported in Fig. 1 for the two extreme cooling rates investigated in the present work. Calculated thermal stress is also reported using the conventional relation which holds when the oxide film is thinner than the metallic substrate. The figure clearly shows that the residual stresses increase with the oxidation temperature whatever the experimental conditions. This behaviour is directly related to the evolution of the thermal stress which is linked to the temperature drop on cooling. However, the measured residual stresses are systematically weaker than the calculated thermal stresses which implies the activation of stress release mechanisms in the ceramic films either during the isothermal step and/or on cooling. In addition, for each considered temperature, the stress magnitude variation is the same for the cooling rate and the oxidation time: a large increase with the cooling rate is observed compare to the oxidation time which may correspond to the effect of the stress build up. The influence of the cooling rates is significant, in particular for the shortest oxidation time of 3 h. The lower is the cooling rate, the more important seems to be the stress release mechanism which is compatible with a creep mechanism activated at high temperature. This effect is more visible after 3 h upon oxidation because it corresponds to the maximum stress amplitude initially reached during the stress



**Fig. 1.** Evolution of the compressive residual stress vs oxidation temperature for different oxidation times and cooling rates: (□) 3 h oxidation, cooling rate of 80 °C/min; (○) 3 h oxidation, cooling rate of 500 °C/min; (△) 18 h oxidation, cooling rate of 80 °C/min and (▽) 18 h oxidation, cooling rate of 500 °C/min. Calculated thermal stresses are also reported (▷).

dynamic evolution at high temperature, e.g. the growth stress [28]. Thus, a maximum of elastic energy is induced which has to be release on cooling. Let us notice that the difference between the calculated thermal stress and the measured residual stress reduces with temperature. An interpretation could be that the stress release mechanism seems to be less efficient at 1000 °C even if it operates at all the considered temperatures in this study. This could be also related to the obtained chromia grain size which is the largest for this temperature.

Thus, from the above residual macroscopic stress determination and the previous work giving the growth stress evolution and modelling for such chromia films on Ni–30Cr alloys, stress release by diffusion-creep seems to be promoted. Now, in order to evidence this phenomena, sample surface morphology features are investigated by AFM, and the influence of the oxidation conditions and the cooling rate, is also considered for comparison with the previous macroscopic stress behaviour. In the case of bulk ceramics or metals, the grain boundary sliding mechanism has already been evidenced and is then well documented. It corresponds to the relative displacement of a grain with respect to its neighbours, without any rotation along a direction that belongs to the average grain boundary plan. Such a sliding needs a geometrical deformation of the boundary or the boundaries network with localised material transport [18–22]. The microscopic strain associated to this sliding can be calculated by using the relation:

$$\varepsilon_{gbs} = \Phi \left( \frac{v_{eff}}{L} \right) \quad (1)$$

where  $\varepsilon_{gbs}$  represents the vertical component of the strain associated to the grain boundary sliding mechanism, and

$$v_{eff} = v - v_0 \quad (2)$$

$L$  is the grain size; in fact the apparent grain size is considered here.  $\varphi$  is a geometrical constant which can be taken equal to 1.4 as it was previously done for bulk alumina,  $v_0$  is the mean vertical offset between adjacent grain before sliding, e.g. before creep initiation, and  $v$  is the mean vertical offset after sliding. In the present case, the thermally grown oxide must be considered as a thin ceramic film supported by a metallic substrate. Since the grain size/film thickness ratio is smaller than a critical value [18], the strain is then transmitted through the entire film thickness. Taking into

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