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Density Measurements after Tensile and Creep Tests on Pure and Slightly Oxidised Aluminium

C. BEGHI, C. GEEL, G. PIATTI
Metallurgy and Ceramics Division, CCR, Euratom, Ispra, Italy

In order to investigate the influence of dispersed oxide particles on crack-formation in Al-Al_2O_3 alloys, series of tensile tests (20 to 550° C) and creep tests (450° C) were carried out on pure Al and on Al-0.7 wt% Al_2O_3 specimens. Density measurements performed on broken samples showed no changes in pure Al but considerable decreases in the slightly oxidised Al. The observed influence of oxide is strong for the small percentage if compared with the behaviour of Al-Al_2O_3 alloys with much higher oxide contents.

In previous work [1–3] the influence of temperature on strain-induced cavitation in Al-Al_2O_3 (SAP) alloys was studied after tensile and creep tests. At high temperature a high rate of crack-formation was found. It was observed [1] that the extent of cavitation as measured by a density change, increased slightly with increasing alumina content (from 4 to 14 wt % Al_2O_3), following an irregular trend at different temperatures. In the present work comparative experiments were carried out on pure aluminium and slightly oxidised aluminium.

The materials used were obtained from high purity aluminium ingots (Al 99.99%) extruded into bars of 20 mm diameter (temperature 500° C, extrusion ratio $\simeq 12$). Some of these bars were utilised for the pure Al samples: average measured density 2.6984 g cm^{-3}. For the preparation of the oxidised material the remaining bars were swaged to a diameter of 2 mm, and oxyacetylene sprayed in air to obtain a slightly oxidised powder. This powder (dimension of particles $< 200 \mu m$) was subsequently hot-pressed and extruded (temperature 500° C, extrusion ratio $\simeq 12$) into bars of 20 mm diameter. Average measured density for this oxidised material: 2.6946 g cm^{-3}.

Chemical analysis showed the sprayed material to have an oxide content of 0.70 wt %. The theoretical density of this material cannot be stated because of uncertainty of the density of the alumina. The oxide was present in the η form (ALCOA denomination) as determined by X-ray diffraction on the alumina extracted from the matrix; the method is described in [4]. The alumina powder, after extraction, was also examined by electron microscopy; the morphology was similar to that of aluminium oxide extracted by the same method from SAP alloys.

The tensile and creep specimens for both the oxidised and pure aluminium material had a gauge length of 50 mm with diameter 10 mm. For both materials two series of experiments were carried out:

(a) Tensile tests at different temperatures from 20 to 600°C; cross-head speed of 3 mm min^{-1} (strain rate 1.0×10^{-3} sec^{-1}).

(b) Accelerated creep tests with constant load at 450° C at different levels. Density measurements were carried out by the immersion technique according to the method described in [2]. Microscopical examination was carried out on several specimens in order to follow eventual grain growth during the experiments.

In fig. 1 the ultimate tensile strength and the total elongation are plotted against the temperature. The results for the total elongation are rather dispersed but to a first approximation it is possible to say that the elongation for pure aluminium increases with increasing temperature while the elongation for the oxidised material decreases, as in SAP alloys [5]. The results of the creep tests are shown in fig. 2 where the applied stresses and the total elongation are plotted against time to rupture .The good linear dependence of the stress on the logarithm of the time to

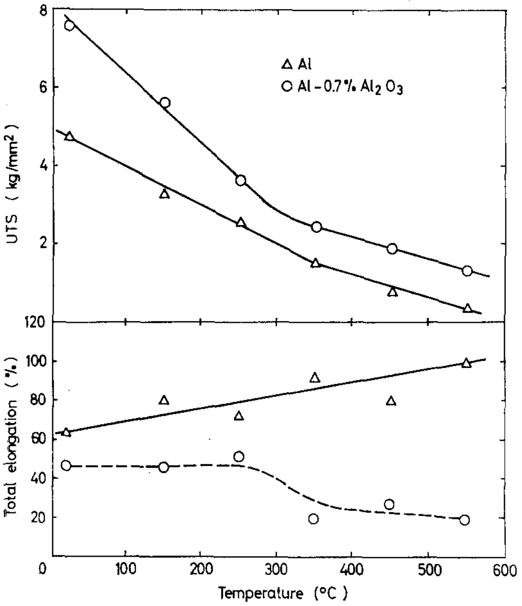

Figure 1 The ultimate tensile strength and the total elongation as a function of temperature for the pure Al and Al-0.7% Al_2O_3. (Strain rate: 1 × 10^{-3} sec^{-1}).

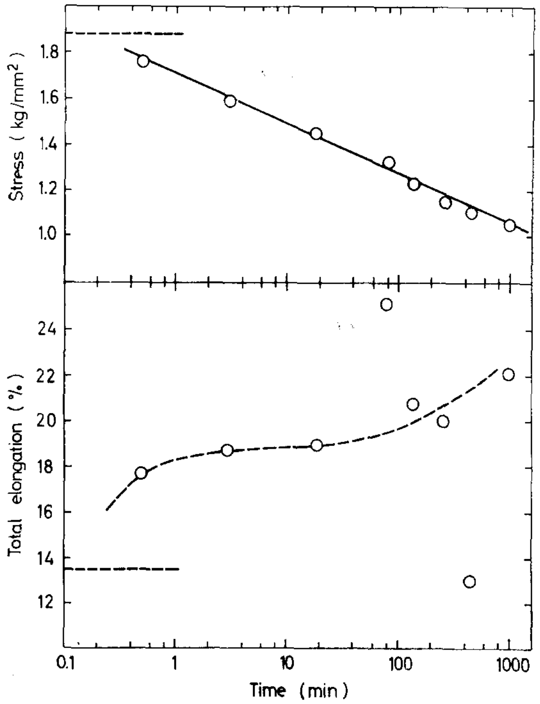

Figure 2 Stress and total elongation as a function of time to rupture (creep test at 450° C) for the Al-0.7% Al_2O_3. The dashed lines indicate the values found in a tensile test executed on a sample taken from the same lot of material utilised for the creep tests. This was a different batch from that used for the test in fig. 1.

rupture is unexpected, considering the high scatter of results usually found in creep tests on SAP alloys [3].

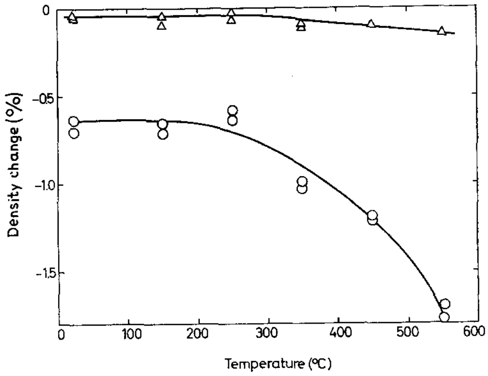

Figure 3 The density change after tensile test as a function of temperature for Al-0.7% Al_2O_3. ○, rupture zone; △, uniform deformation zone.

No density changes were found in the tensile and creep test specimens of pure aluminium, but there was a considerable density decrease in the rupture zones of the oxidised aluminium specimens. Fig. 3 shows this decrease as a function of test temperature for the tensile specimens. Up to *ca.* 250° C the density change remains practically constant but at higher temperatures it increases rapidly. The elongation (fig. 1) follows the same trend. In the zones of uniform deformation of these specimens only a slight variation in density was found. In order to see the local variation of density with deformation, specimens broken at 450° C were cut into small samples of *ca.* 0.5 g. The density change in these samples is plotted against the reduction in area of the section from which they were cut in fig. 4. For comparison a

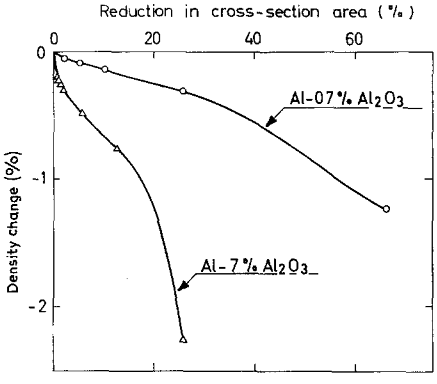

Figure 4 The density change of Al-0.7% Al_2O_3 and Al-7% Al_2O_3 alloys strained by tensile deformation at 450° C, plotted against reduction in area.

curve for 7% Al_2O_3, taken from [2], is also shown.

The variation of density with time to rupture in creep at 450° C is shown in fig. 5 for the oxidised aluminium. For times to rupture up to 4 min, the density change remains constant and corresponds to the values found in the normal tensile tests at 450° C. However, at longer times high reductions in density were found. A density decrease of 7.5% was measured after 1000 min.

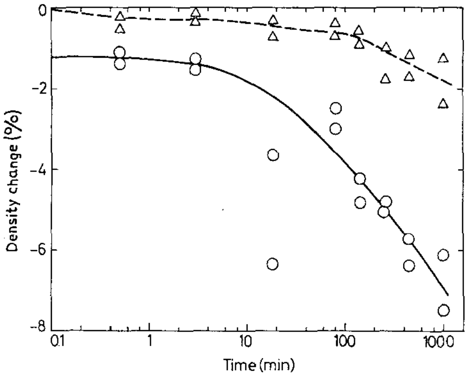

Figure 5 The density change as a function of the time to rupture (creep tests at 450° C) for Al-0.7% Al_2O_3. ○, rupture zone; △, uniform deformation zone.

Metallographic examination showed, as expected, a considerable grain growth in the pure aluminium specimens, especially after the longer creep experiments. No grain growth was detected in the oxidised aluminium.

The experimental results show clearly that even a small percentage of oxide in aluminium causes a considerable density decrease during tensile and creep tests, under conditions where no density changes are found in the pure metal. This is in agreement with the hypothesis that second phase particles are the main sources of cavitation in Al-Al_2O_3 alloys [2]. Fig. 6, which reports in addition to the new data on Al-0.7% Al_2O_3, data for 4, 7 and 14% Al-Al_2O_3 alloys [1], shows that at 450° C the density variation is not proportional to the oxide content, but changes rapidly at low concentrations and remains practically constant at high oxide values. The oxide distribution is generally intergranular in our alloys, while in the SAP alloys it is also intragranular. Because the cavitation is presumably associated with the particles in grain-boundaries, the density variation is not very different passing from an oxidised aluminium to a SAP. Nucleation at the interfaces of the intragranular particles does not influence cavitation because the growth rate there is lower than that at the interfaces of the intergranular particles. The difference in density variation is presumably more related to the difference in grain size than to the difference in total oxide content. It has been shown [7] that in the SAP alloys void nucleation is more important than the void growth. Presumably also in Al-0.7% Al_2O_3 alloy the important effect of the oxide is in controlling cavity nucleation.

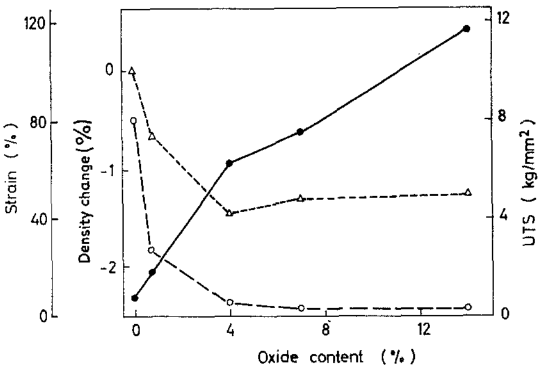

Figure 6 Density change in rupture zone, ultimate tensile strength and strain as a function of oxide content at 450° C (tensile tests with strain rate 1 × 10^{-3} sec^{-1}). △, density change; ●, ultimate tensile strength; ○, strain (total rupture elongation).

A detailed analysis of the density changes caused by the different oxide contents is complicated by the fact that not only the difference in oxide content, but other parameters as well play a rôle in cavitation (deformation, stress to

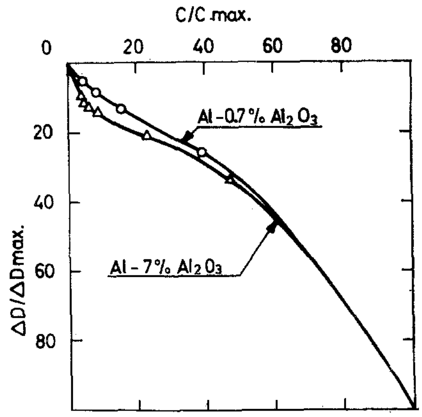

Figure 7 Density change ratio (%) as a function of reduction in area ratio (%); tensile tests performed at 450° C.

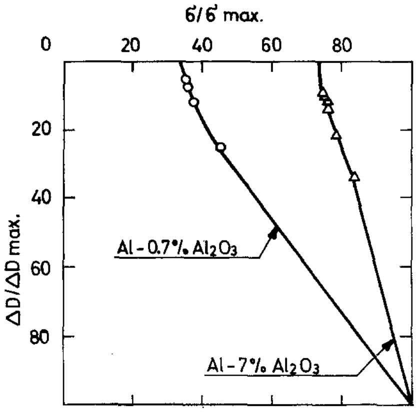

Figure 8 Density change ratio (%) as a function of stress ratio (%); tensile tests performed at 450° C.

rupture etc). In order to facilitate this analysis, density change values relative to the change in density in the rupture zone ($\Delta D/\Delta D_{max}$) are plotted against relative values for cross-section reduction (C/C_{max})* (fig. 7) and the relative values for stress (σ/σ_{max})* (fig 8). The comparison of the two figures leads to the suggestion that the phenomenon is controlled directly by the deformation process and not by the stress: the curves in fig. 7 are practically coincident.

For other materials, as for instance Magnox Al 80, Ni-0.1 at.% Pd [6] and SAP 7 wt % Al_2O_3 [7], evaluation of the density change during creep indicates that the cavitation is also governed by the deformation process.

Acknowledgement

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*C is the reduction in area for a particular section of the tensile specimen.
C_{max} is the reduction in area in the rupture zone.
σ is the final stress in each section of the tensile specimen (the rupture load divided by the cross-sectioned area).
σ_{max} is the final stress in the rupture zone.