ea 1989/04

Scripta METALLURGICA Vol. 23, pp. 1595-1598, 1989 Printed in the U.S.A.

HIGH TEMPERATURE Al-Al_2O_3 ALLOYS WITH A COARSE ELONGATED GRAIN STRUCTURE

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(Received July 5, 1989)

Introduction

In view of the demand for new light-weight materials in the temperature range 300 - 500 °C, great efforts are presently being made to increase the temperature capability of aluminium alloys, e.g. (1). A promising route is the development of aluminium strengthened by thermally stable intermetallic or non-metallic dispersoid particles. This aim is currently tackled by the application of two different processing methods: rapid solidification of supersaturated aluminium alloys (e.g. Al-Fe-Ce (2), Al-Fe-V-Si (3)), which leads to fine intermetallic precipitates; and the incorporation of carbide and oxide dispersoids by mechanical alloying and related techniques (for a recent overview see (4)). This paper is concerned with an aspect of alloy development by the latter technique.

We focus on the effect of grain size on the high-temperature strength of dispersion-strengthened aluminium. While much attention has been paid in the literature to the dispersoid particles themselves, the aspect of grain size optimization in such alloys seems to have received only little attention. In fact, all current dispersion-strengthened aluminium alloys produced by mechanical alloying techniques exhibit a small grain size of the order of 1 μm. This is somewhat surprising as it is well known that the presence of grain boundaries generally impairs the high-temperature strength because of grain-boundary sliding, diffusional creep and/or premature intergranular failure. This is particularly true of dispersion-strengthened alloys, e.g. ODS superalloys (5) and ODS ferritic alloys (6). To compensate for the large strength differential between grain boundaries and grain interiors in such alloys, grain coarsening treatments - sometimes by gradient annealing (7,8) - are necessary for exploiting the full potential of dispersion strengthening at high temperatures. The application of such treatments to dispersion-strengthened aluminium alloys however does not appear to have been reported.

This paper will describe the grain coarsening behaviour in a dispersion-strengthened aluminium alloy and some preliminary effects on the short-term strength. A more detailed analysis will be published elsewhere (9).

Experimental

The experiments were performed with an Al-1 wt % O alloy (tradename DISPAL (10)). This materials is produced by "reaction milling" and extruding aluminium powder (11). The material was provided by Sintermetallwerk Krebsöge and Erbslöh Aluminium with a circular cross-section (diameter 12 mm). A typical TEM micrograph of the as-received alloy is given in fig. 1, where the Al_2O_3 dispersoids are visible as grey spots. Typical particle diameters are 30 nm, characteristic spacings 160 nm (12). The theoretical volume fraction of the dispersoids in this alloy amounts to 1.86 %. The alloy did not contain deliberately added carbon, and hence practically no carbide dispersoids. Typical grain dimensions are 4 μm in the extrusion direction and about 2 μm in the transverse direction, resulting in a grain aspect ratio of about 2.

Recrystallization studies were conducted by combinations of reductions and annealing under various conditions. The pre-deformation strains ranged from φ = 1.75 (reduction to 5 mm) to 2.77 (3 mm), annealing temperatures were varied between 440 and 600 °C. In order to optimize the recrystallization response, stepwise reductions were also performed.

To characterize the success of the recrystallization treatments, polished sections were etched electrolytically following the procedure described by Barker (13). Best results were obtained at 20 V, with an etchant of

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the following composition: 0.7 g H_3BO_3, 10 ml HF (40 %) and 200 ml H_2O (dist.). Due to the deposition of an anisotropic interference layer, observation in polarized light gave strong colour differences between adjacent grains. The grain dimensions were usually measured on longitudinal sections, while the fraction of recrystallized volume was determined on transverse sections.

First mechanical tests at temperatures up to 550 °C were performed on cylindrical specimens of fine-grained (as-received) and recrystallized material with dimensions 8 mm diameter by 16 mm long and 5 by 10, respectively. Testing was in compression, with a constant strain rate of 10^{-3} s^{-1}.

Results and Discussion

Recrystallization

Fig. 2 shows the effect of pre-deformation strain and of annealing temperature on the fraction of coarse-grained volume. It is apparent that the recrystallization response is generally poor below 500 °C, while at 600 °C almost complete recrystallization could be achieved irrespective of pre-strain. In the intermediate temperature range the response depends on the amount of pre-strain, and is poorest after medium strain ($\varphi = 2.2$). Temperatures above 600 °C are not advisable because of the evolution of internal gases which severely damage the material.

The time-dependence of the recrystallization process is depicted in fig. 3. Again the strong influence of the annealing temperature is apparent. At 600 °C, recrystallization is completed in less than 5 minutes, regardless of pre-strain, whereas the process is very sluggish at 480 °C.

In the course of the experiments it was found that this simple recrystallization treatment involving a single pre-strain followed by annealing did not give reproducible results in other batches of the same material. To remedy this situation, pre-straining in two steps, with an intermediate annealing treatment, were tried out. Best results were obtained by reduction from 12 to 6 mm, followed by annealing at 500 °C for 30 minutes, a further reduction to 5 mm and a final heat treatment at 600 °C for 5 minutes. By this method, complete recrystallization to coarse grains could be achieved reproducibly in all available batches.

The resulting coarse grain structure is shown in fig. 4. Typical grain dimensions are 5 mm by 0.3 mm, with a grain aspect ratio of about 15. It is noted that the coarse grain structures produced in this Al-Al_2O_3 alloy exhibit a striking similarity with the grain structure of dispersion-strengthened superalloys after recrystallization (7,8,14). The kinetics of the process suggests that the mechanism of grain coarsening is also similar, i.e. secondary recrystallization (8,15).

For comparison, attempts were made to recrystallize aluminium alloys which in addition to oxides also contained carbide dispersoids (volume fraction 6 %). Noticeable grain coarsening could not be obtained in this material.

Compressive Strength

The compressive strengths of fine-grained and coarse-grained material, measured at a strain rate of $\dot{\varepsilon} = 10^{-3}$ s^{-1} as a function of temperature, are compared in fig. 5. At room temperature the strength of the fine grain (112 MPa) lies well above that of the coarse grain (82 MPa). The difference can be attributed to grain-boundary strengthening under the assumption that the Hall-Petch constant is similar as in pure Al (12). With increasing temperature the following important observation can be made: Although the beneficial grain boundary effect decreases, the fine grain is always superior to the coarse grain up to the highest test temperature of 550 °C.

The fact that a "cross-over" in strength does not occur is in marked contradiction with similar measurements on other dispersions-strengthened alloys: Both a ferritic ODS alloy, Incoloy MA 956 (16), and an ODS superalloy, IN 738 + Y_2O_3 (17), exhibit an "equi-cohesive" temperature at which the strength of the fine grain begins to drop below that of the coarse grain. In both cases, this cross-over occurs almost exactly at half the absolute melting point as would be expected on the basis of increasing thermal activation of grain boundary processes. By contrast, no such behaviour is apparent in our alloy at 0.5 $T_m \approx 200$ °C.

The reasons for this surprising behaviour are not fully understood. First results indicate that the texture, which is strongly developed in our fine-grained alloy but is absent in other dispersion-strengthened alloys after extrusion, may play a decisive role. Further work along these lines is in progress. In addition, testing under tension and creep testing will have to complement these results. It is to be expected that under these conditions the grain boundaries will have a more detrimental effect than under compression.

Conclusions

Our results indicate that dispersion-strengthened aluminium intended for use at high temperatures can be recrystallized to a coarse grain structure in a similar way as other dispersion-strengthened alloys, provided that the dispersoid content is not too high. A special two-step process has been developed which allowed complete grain coarsening by secondary recrystallization to be achieved. First mechanical tests show however that contrary to expectations the coarse grain is weaker (in compression) than the fine grain up to the highest test temperature of 550 °C. It thus appears that a coarse grain structure may not be required for achieving high strength in dispersion-strengthened aluminium alloys at high temperatures.

Acknowledgement

The authors are grateful for the supply of material by Dr. Arnhold, Sintermetallwerk Krebsöge, and Dr. Brockmann, Erbslöh Aluminium. They acknowledge partial funding of this project by the German BMFT under project number 03M00 10E4.

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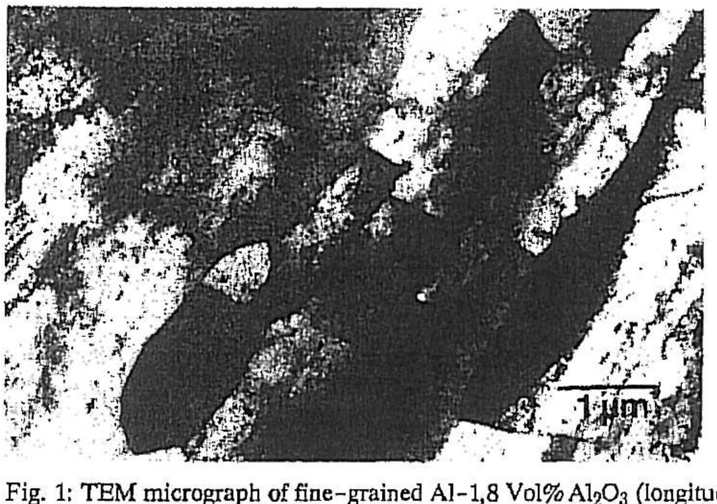

Fig. 1: TEM micrograph of fine-grained Al-1,8 Vol% Al_2O_3 (longitudinal direction), showing the distribution of Al_2O_3 dispersoids.

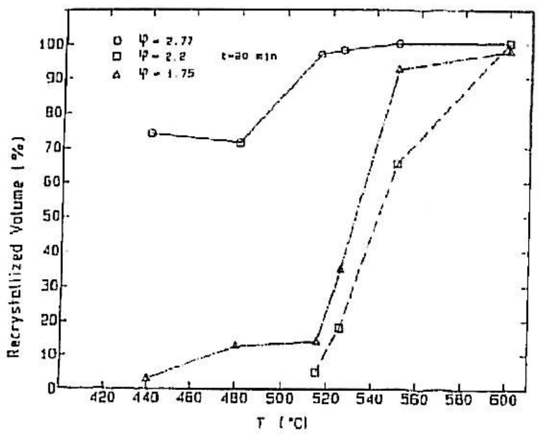

Fig. 2: Volume fraction of recrystallized coarse grain as a function of annealing temperature for different degrees of pre-straining.

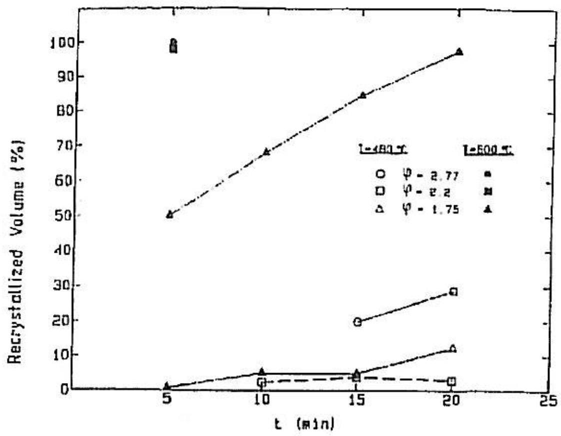

Fig. 3: Time dependence of the grain coarsening process at 480 °C and 600 °C for various degrees of pre-straining.

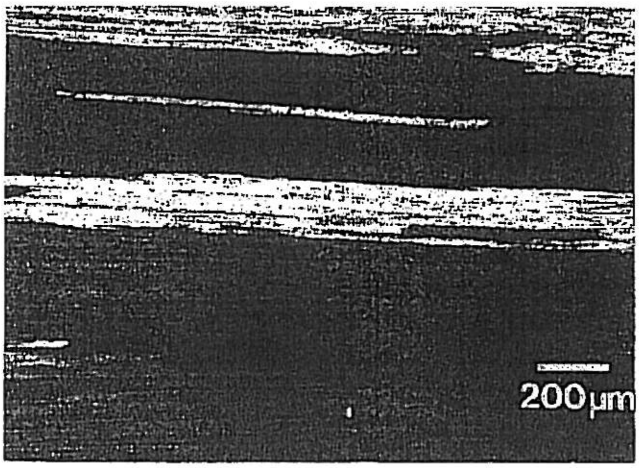

Fig. 4: Coarse elongated grain structure of Al-1.8 Vol% Al_2O_3 obtained by recrystallization.

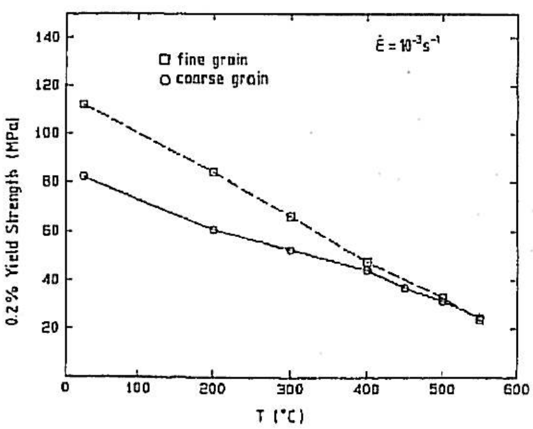

Fig. 5: Compressive yield strength ($\sigma_{0.2}$) of Al-1.8 Vol% Al_2O_3 in fine-grain and coarse-grain condition as a function of temperature ($\dot{\varepsilon} = 10^{-3}\ s^{-1}$).