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Cavitation in alloy steels during superplastic deformation

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A study of cavitation during superplastic tensile straining of two microduplex steels has been made using density measurements and quantitative optical metallography. The steels were of basically similar composition with the exception of a trace addition of boron made to one alloy. During deformation cavities formed at α/γ boundaries and matrix-carbide interfaces; the growth and coalescence of these cavities led to failure. Density measurements showed that the extent of cavitation increased with increasing strain and decreasing strain-rate, but the level of cavitation was reduced by the presence of boron. A time dependence of overall void volume of 1.4 to 2.0 was observed. Quantitative metallographic studies of the nucleation and growth contributions to the overall rate of void formation showed that boron inhibited each of these processes. However, both the nucleation rate and the magnitude of the time exponent of void volume increase suggested that a substantial number of voids grew from pre-existing nuclei which were probably present as non-coherent carbide-matrix interfaces.

1. Introduction

More extensive use of the superplastic potential of metals depends on (a) extending the range of superplastic behaviour to higher strain-rates approaching those associated with conventional forming processes, and (b) inhibiting any process which could lead to premature failure during superplastic flow. In creep, failure often results from the nucleation, growth and coalescence of grain-boundary cavities, and it has recently been observed that a similar sequence of events may lead to failure during the superplastic tensile deformation of steels [1]. The cavitation process reflects the inability of accommodation processes to maintain grain-boundary cohesion during grain-boundary sliding, which is an important mode of superplastic deformation. To improve the resistance of superplastic materials to grain-boundary cavitation, the basic mechanisms involved must be understood and quantified. Although cavitation might not result in failure during superplastic forming, it may have a detrimental effect on the service behaviour of superplastically-shaped components.

The present investigation has involved a study of the cavitation process in superplastic alloy steels, using densitometric and quantitative metallographic techniques. Work has been carried out on an alloy (K1970) of composition Fe–4 wt % Ni, 3 wt % Mo, 1.6 wt % Ti, 0.03 wt % C, and also on a further alloy (K2261) of identical composition, containing a trace addition of boron (60 ppm).

2. Experimental

The steels examined were supplied by the BSC Corporate Development Laboratory as hot-rolled bar of 12.7 mm diameter. The K1970 alloy which was developed by Smith and Ridley [2] to show good superplastic behaviour, had a duplex $\alpha + \gamma$ structure in the temperature range 850 to 1000° C, with a phase volume ratio of approximately 1:1 in the range 900 to 960° C. Both alloys were cold swaged to 7.9 mm diameter bar and tensile specimens of circular section were machined. The specimens (shown in profile in Figs. 2 and 3) were 3.80 mm in diameter and had a gauge length of 10 mm. Recrystallization of the specimens was carried out in the tensile rig prior to straining at the temperature of the test, 900 or 960° C. Measurements of the mean linear intercept of the microduplex $\alpha + \gamma$ structures produced were 5 and 7 µm, respectively. Tensile straining was carried

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out *in vacuo*, in equipment attached to an Instron testing machine.

In order that both steels might be characterized, the strain-rate sensitivity, *m*, was determined using the method of Backofen *et al.* [3], the crosshead velocity being cycled progressively through values between 0.5 and 0.0005 cm min^{-1} at 960° C. A further comparison of the two steels was obtained by means of constant crosshead velocity tests to failure, at 900 and 960° C, when specimens were strained at various strain-rates in the region of maximum strain-rate sensitivity, *m*.

Specimens for density measurement were strained at a constant crosshead velocity to a pre-determined strain. No measurements were made on specimens strained near to the point of failure. After straining, the parallel-sided gauge lengths were cut from the screw-threaded gauge heads. The screw-thread was machined from one of the gauge heads, producing a smooth cylindrical standard, suitable for accurate density work. Both the gauge head ($\sim$ 4.0 g) and the gauge length ($\sim$ 0.7 g) were electropolished to produce a bright finish. Density measurements were made at constant temperature by hydrostatic weighing in ethylene dibromide, using a Mettler microbalance.

By measuring the size distribution of voids in cavitated specimens, it is possible, using methods first developed by Scheil and Lange-Weisse [4] to determine both void nucleation and void growth rates. Information was obtained by examining selected density specimens, at various stages in the cavitation process. Each specimen was mounted longitudinally, ground to the gauge length diameter, and then carefully polished to a 1/10 μm finish. Examination of the polished surfaces using the scanning electron microscope revealed minimal "edge-effects" at the void-polished surface interface. Measurement of the void size distributions was attempted using (i) the Quantimet 720 Image-analysing Computer, and (ii) the Zeiss TGZ3 Particle-size Analyser.

3. Results

The relationship between stress and strain-rate for each steel at 960° C is shown in Fig. 1. It can be seen that the presence of boron affects the deformation characteristics, reducing the maximum *m* value from 0.53 to 0.37, and raising the stress level for a given strain-rate. During constant crosshead velocity tests at 900 and 960° C, the boron steel, under identical conditions, tended to produce lower elongations, as one might expect from the lower *m* value, although the highest elongation observed in the present work (634% at 0.01 cm min^{-1}) was obtained for the latter alloy, at a low strain-rate. Both steels showed a rapid reduction in the elongation values achieved at higher strain rates, consistent with the reduction in *m* values. Figs. 2 and 3 show the fractured specimens of both steels after constant velocity straining at 900° C, at various strain-rates.

The extent of cavitation is shown in Fig. 4 in a polished longitudinal section of a K1970 (boron free) specimen strained at 0.1 cm min^{-1} to 350% elongation at 960° C. It can be seen that the cavities are fairly uniformly distributed across the specimen diameter, but tend to lie in groups or "chains", orientated in the direction of the applied stress. Many cavities are associated with TiC particles present in the microstructure.

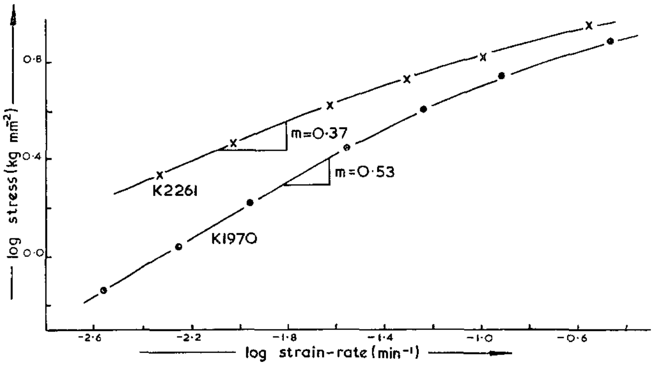


Figure 1 Logarithmic plots of stress versus strain-rate for the boron-free (K1970) and boron-doped (K2261) steels at 960° C.

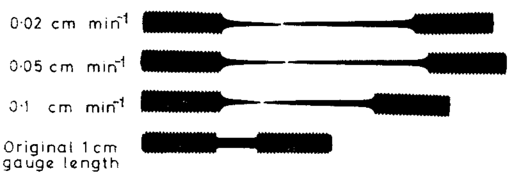


Figure 2 Fractured tensile specimens of the boron-free steel (K1970) strained at various rates at 900° C.

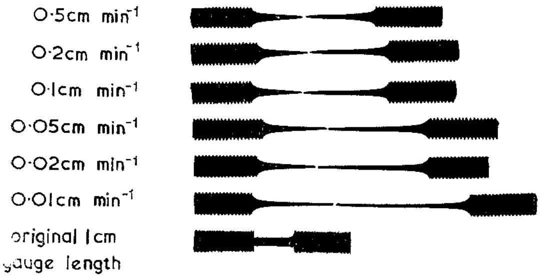


Figure 3 Fractured tensile specimens of the boron-doped alloy (K2261) strained at various rates at 900° C.

Examination of the failed specimens using the SEM revealed cavities in the region of the fracture surface. Fig. 5 illustrates a specimen strained to failure (442%) at 0.05 cm min^{-1} and 960° C. The premature nature of the failure, due to growth and interlinkage of cavities, is demonstrated by the parallel sides of the specimen near the fracture surface.

To examine the cavitation process in greater detail, specimens were strained at 960° C, at 0.1 and 0.05 cm min^{-1}, to pre-determined strains. The results obtained from density measurements on these specimens are shown in Fig. 6. It can be seen that (i) the extent of cavitation increases with increasing deformation (ii) a decrease in strain rate leads to an increase in the level of cavitation, and (iii) boron reduces the amount of cavitation.

Using the Quantimet, measurements were made of the total volume of voids, and the void size distribution, on four longitudinal polished sections of K1970 strained to varying degrees at

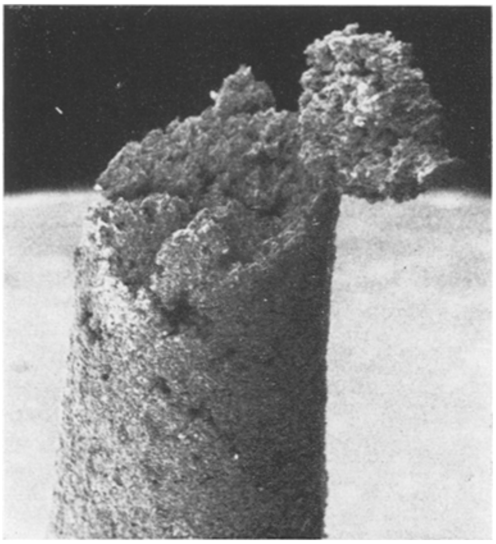


Figure 5 Scanning electron micrograph of the fracture surface of the boron-doped alloy (K2261) strained to failure at 0.05 cm min^{-1} and 960° C.

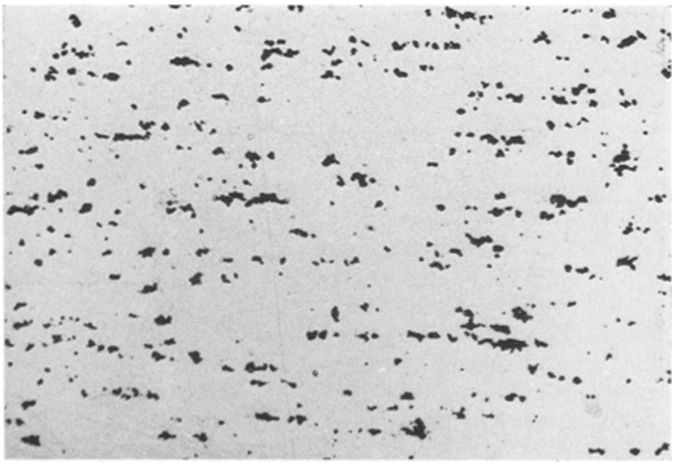
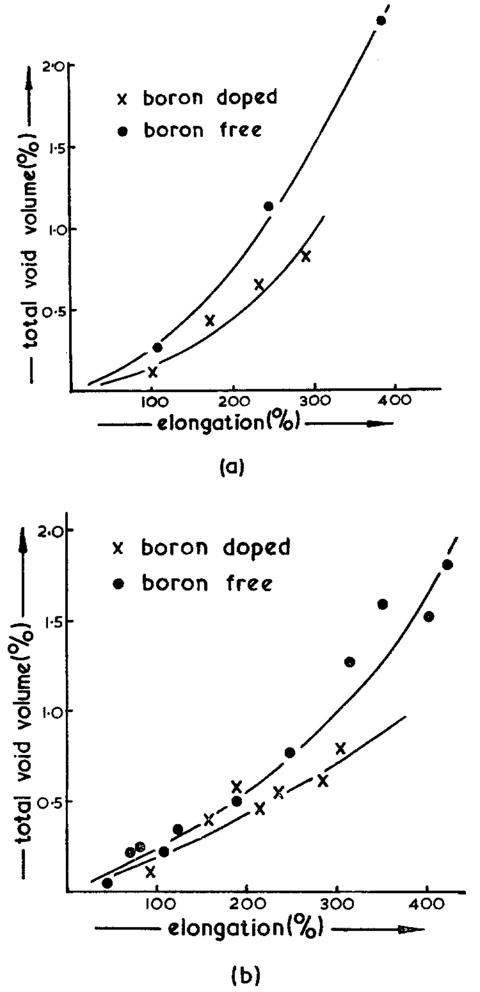


Figure 4 Polished longitudinal section of the boron-free alloy (K1970) strained to 350% at 960° C and 0.1 cm min^{-1}. The stress direction is horizontal (× 105).



(a)

(b)

Figure 6 Density changes during superplastic straining at 960°C at (a) 0.1 cm min^{-1} and (b) 0.05 cm min^{-1}.

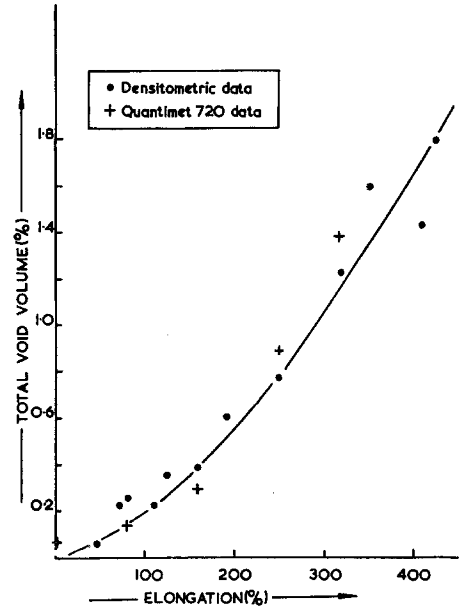


Figure 7 Comparison of the density and Quantimet techniques for the measurement of void volume in the boron-free steel (K1970) strained at 960°C and 0.1 cm min^{-1}.

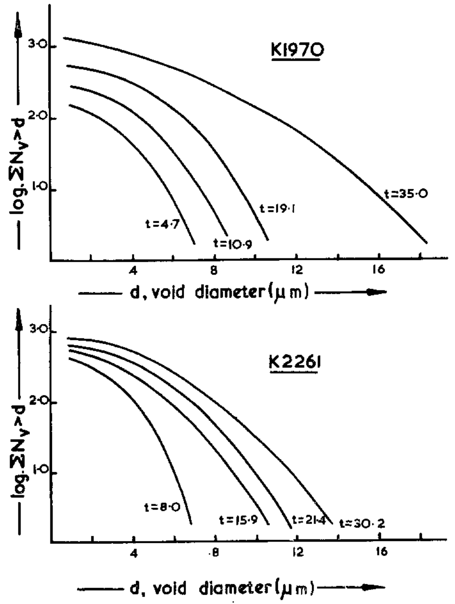


Figure 8 Inverted cumulative distribution curves for the two alloys deformed at 960°C at 0.1 cm min^{-1}.

960°C at 0.1 cm min^{-1}. Fig. 7 shows a comparison of density measurements with the values obtained on the Quantimet. In addition to the estimation of total void volume, the Quantimet has also been used to obtain some measure of the void size distribution. Although the results compared favourably with estimates obtained using the Zeiss Particle-size Analyser, the latter method was used to obtain subsequent data. Difficulties associated with the Quantimet used involved (i) the inability to store information in more than seven size groups, (ii) the lack of resolution of smaller voids, and (iii) the need to scan a large area in order to obtain reproducible data.

The observed void size distribution, corrected by a modified Scheil analysis, to give a true void size distribution, was obtained in terms of inverted cumulative distribution curves, as shown in Fig. 8. The void diameter, d, was plotted against the log of the sum of the number of voids having a diameter greater than that diameter, $\sum N_V > d$. These curves enable the

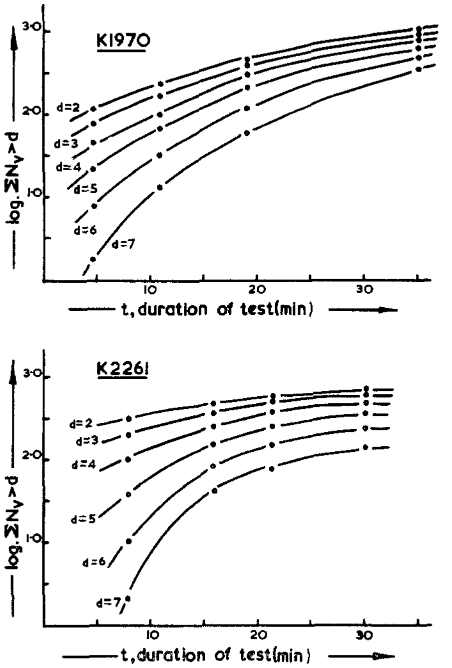


Figure 9 Plots of log $\sum N_V > d$ versus deformation time, t, constructed from Fig. 7 for various values of d.

process of cavitation in both steels, K1970 and K2261, to be compared in terms of the component processes of void nucleation and void growth (Figs. 9 and 10 respectively) using the procedures outlined by Brown and Ridley [5]. The slope of the curves in Fig. 9 represents the rate of void nucleation, and Fig. 10, the rate of void growth.

4. Discussion

It has been observed that cavitation occurs during the superplastic deformation of a low alloy steel and that cavity growth and coalescence is ultimately responsible for failure.

Density measurements have shown that the extent of cavitation increases with increasing strain and decreasing strain rate. The work of Smith and Ridley [6] on the superplastic duplex stainless steel, IN744, supports these observations, and in addition, has shown that the level of cavitation increases with increasing temperature, and to a lesser extent, with increasing grain size.

Since the specimens are strained at a constant crosshead velocity, so that nominal strain is proportional to time, the results shown in Fig. 6 may be presented on a log V versus log t basis, assuming an equation of the form

$$V = Kt^n \qquad (1)$$

where V = total void volume, K = constant, t = test duration in minutes, and n is the time exponent. Fig. 11 illustrates log V versus log t plots for both steels at two strain-rates; the curves show that overall void volume is proportional to $t^{1.4-2.0}$.

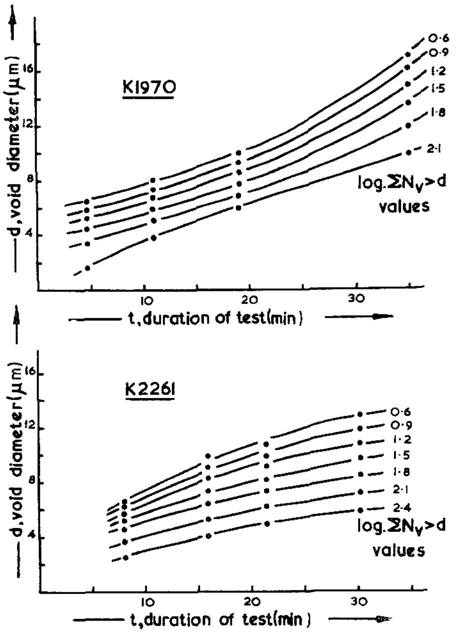


Figure 10 Plots of void diameter, d, versus deformation time, t, constructed from Fig. 7 for various values of log $\sum N_V > d$.

As suggested by Smith and Ridley [6], one way of interpreting the significance of the time exponent is to make reference to the Avrami equation [7], which is widely used to analyse kinetic data in phase transformation and precipitation studies. For small volume fractions transformed, the Avrami equation is identical in form to that used above to analyse the experimental data, and by analogy with the transformation situation, deductions may be made concerning the physical processes associated with cavitation, from the values of the time exponent.

Christian [8] has given values of n for a range of transformation conditions. Values between 1.5 and 2.5 are consistent with diffusion-controlled growth of precipitates, of all shapes,

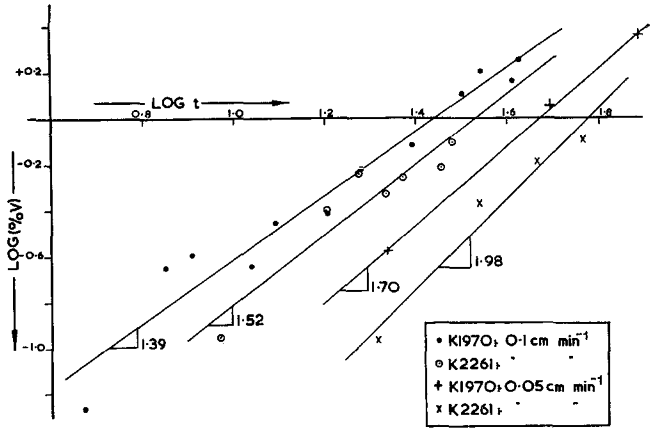


Figure 11 Logarithmic plots of void volume versus deformation time for the two alloys deformed at 0.1 and 0.05 cm min^{-1}.

growing from small dimensions at a decreasing nucleation rate, with the limiting values of 1.5 and 2.5 corresponding to zero and constant nucleation rates, respectively. In the present work, time exponents in the range 1.4 to 2.0 have been observed. The value of 1.4 is close to that of 1.5, also given by the Hull and Rimmer [9] analysis of void growth during creep, and suggests that no true nucleation events are occurring under these conditions. Thus, growth may be taking place at pre-existing nuclei, which are probably provided by TiC inclusion-matrix interfaces in the steels.

It would be desirable to resolve quantitatively the overall kinetics of void formation, into the component nucleation and growth events. However, it is difficult to determine whether the so-called nucleation, involves true nucleation, or includes "nucleation" at the non-coherent particle-matrix interfaces as suggested above. From Fig. 9 it may be seen that in both steels the rate of nucleation tends to decrease as the test proceeds, as potential nucleation sites become exhausted. Further evidence for the presence of pre-existing nuclei in both steels may be obtained from Fig. 9 by extrapolating the curves to zero time. For other than cavities of large diameter the curves intercept the $\sum N_V > d$ ordinate and indicate that many nuclei having a range of sizes exist at zero time, i.e. before deformation starts. These nuclei are probably located at carbide-matrix interfaces. A comparison of the two steels in Figs. 9 and 10 reveal that the rates of void nucleation and growth are generally lower in the boron steel, than in the boron free steel, especially during the latter stages of the test.

It is clear that a small addition of boron has a significant effect on both the cavitation and deformation behaviour of the low alloy steels considered. This effectiveness is probably due to the segregation of boron to grain boundaries. Other workers have reported that boron stabilizes grain boundaries [10], retards grain-boundary carbide formation [11], reduces interfacial energy [12], and reduces the grain boundary self-diffusion coefficient, D_{gb} [13], in various systems. Under the test conditions described, the movement of vacancies along the grain boundary to the void is considered an important void growth mechanism. If boron reduces D_{gb} by inhibiting the flow of vacancies to the void, this will decrease the void growth rate as observed. This same type of interaction will inhibit grain-boundary accommodation processes, and produce the observed decrease in *m* value. This interaction of boron atoms with vacancies is considered largely to be responsible for the changes observed in the deformation and cavitation behaviour of the low alloy steel containing boron.

5. Conclusions

1. Tensile studies have been carried out on two microduplex alloy steels, at 960°C. The maximum slopes of the log stress versus log strain-rate plots for these steels were consistent with good superplastic behaviour, although it

was noted that the presence of boron in one of the alloys reduced the maximum strain rate sensitivity, *m*, from a value of 0.53 to 0.37.

2. During straining, cavities were formed at interphase boundaries and at carbide-matrix interfaces. The growth and interlinkage of these cavities was responsible for eventual failure. Although the cavitation process resulted in premature failure of the steels, elongations in excess of 600% were achieved, under optimum conditions.

3. Density measurements showed that the extent of cavitation increased with increasing strain and decreasing strain-rate.

4. A time dependence of overall void volume of 1.4 to 2.0 was observed. The exact value of the exponent of time depended on the strain-rate and material composition.

5. The level of cavitation was reduced by the addition of boron.

6. Interpretation of the time exponents of void volume increase in terms of the Avrami equation, suggests that a substantial number of voids develop from pre-existing nuclei. These nuclei are probably in the form of non-coherent carbide-matrix interfaces.

7. Data obtained by quantitative metallography have been analysed to determine the nucleation and growth contribution to overall rates of void formation, and the indications are that boron inhibits both of these processes. However, quantitative interpretation of data is difficult because of the anomalous effect of pre-existing nuclei on the nucleation rates.

Acknowledgement

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