JOURNAL OF MATERIALS SCIENCE 4 (1969) 195–201

Creep of Austenitic 20% Cr/35% Ni Stainless Steels at Low Stresses

R. LAGNEBORG, R. ATTERMO
Aktiebolaget Atomenergi, Stockholm, Sweden

Received 2 July 1968, and in revised form 13 September

The present work comprises measurements of the secondary creep-rate at different stress levels with rates between about 2×10^{-5} %/h and 10%/h and the grain-boundary sliding at 700° C in two austenitic 20 wt % Cr/35 wt % Ni stainless steels. One alloy was a pure 20 wt % Cr/35 wt % Ni steel, whereas the other contained about 0.5 wt % Ti and 0.5 wt % Al so that it precipitated γ' during creep at 700° C. Special care was taken to assure equivalent microstructure in the specimens and precise creep conditions so as to obtain accurate and reproducible creep-rates. Both materials exhibited decreasing stress-dependence of the creep-rate at low stresses. Neither the stress-dependence of the creep-rate, nor the absolute creep-rate was consistent with diffusion-creep. The amount of grain-boundary sliding was measured separately by means of scribed grid lines on the creep specimens for the pure material at stresses above the "creep yield". The values for the component of the creep-rate due to grain-boundary sliding coincide very well with the extrapolated line of the low-stress branch of the creep-rate/stress curve. All these results taken together suggest that the most likely explanation of the creep yield in 20 wt % Cr/35 wt % Ni steels is the one based upon grain-boundary sliding.

1. Introduction

To make creep testing less time-consuming there is a desire to extrapolate data for rupture time and creep-rate at low stresses from the more readily available data at high stresses. For instance, it is well known that the creep-rate and stress at high temperatures obey a power law, $\dot{\epsilon} = A\sigma^n$, within a relatively large range of creep-rates. The question is, of course, whether such a relation is valid at the low stresses which the material experiences in an actual construction and for which few, if any, experimental data points exist.

An increasing amount of evidence is now becoming available, and it shows that materials creep faster at low stresses than the power law fitted to data at higher stresses predicts. Data for magnesium and zirconium alloys indicate that the exponent, n, in the power law is about 1 at low stresses, whereas it is between 3.5 and 6 at higher stresses [1-5]. This behaviour gives rise to a break, or a *creep yield*, in the stress/creep-rate relation in a double-logarithmic graph.

Jones [3, 4] and Bernstein [5] have proposed that the decreasing stress-dependence of the creep-rate at low stresses and at about 0.6 T_m and even below is due to grain-boundary diffusion-creep, i.e. the transport of matter takes place by diffusion along the grain-boundaries. At somewhat higher temperatures volume diffusion-creep, i.e. where the diffusional transport occurs inside the grains, may account for the decreasing stress-dependence [1-4]. Analytically, grain-boundary diffusion-creep has been formulated by Coble [6]:

$$\dot{\epsilon} = \frac{B}{d^3} \frac{\Omega\sigma}{kT} \omega \, D_{ogb} \exp(-\,Q_{gb}/kT) \qquad (1)$$

where B is a geometrical factor; Ω is the atomic volume; σ is the applied stress; d is the diameter of a spherical grain; ω is the effective width of the grain-boundary; D_{ogb} is the frequency factor for grain-boundary diffusion; Q_{gb} is the activation energy for grain-boundary diffusion.

Jones [3, 4] and Bernstein [5] have shown that this equation may account for the decreasing stress-dependence of the creep-rate at low stresses for magnesium and zirconium alloys. It gives a linear stress-dependence in agreement with the experiments mentioned above and furthermore the calculated creep-rates correlated with the observed. Another finding that has been taken as a strong support for diffusion-creep is the observation of zones denuded of precipitates at grain-boundaries normal to the stress axis in a Mg/0.5 wt % Zr alloy [7]. It was proposed that the zones arose from the diffusional flow of matter. Suggestions have also been made that diffusion-creep may be important for austenitic stainless steels [8] and nickel-base alloys hardened by γ' [9] at temperatures around 0.6 T_m.

Recently the idea of diffusion-creep has been disputed [10]. It was shown that the experimental basis for the linear stress-dependence of the creep-rate has been dubious in some cases. Furthermore, equation 1 contains constants, B, and ωD_{gb}, that are not very well known. Hence, the agreement between observed creep-rates at low stresses and creep-rates calculated from equation 1 may be fortuitous. At present the best support for the diffusion-creep hypothesis seems to be the observations of the denuded zones [7]. However, Raraty has rejected this conclusion [11]. In contradiction to the results by Harris and Jones [1] in a Mg/0.5 wt % Zr alloy, he found no correspondence between the size of the denuded zones and the actual creep-strain in a Mg/1.25 wt % Mn alloy.

Recently it was suggested by Lagneborg [12] from quite general arguments that the observed "breaks" in the stress/creep-rate relation may be explained by an increasing contribution of the grain-boundary sliding to the total creep-strain on lowering the stress. The present work is an attempt to pursue this idea. Therefore, we have carried out high precision measurements of the creep-rate at different stress levels for pure 20 wt % Cr/35 wt % Ni steels at 700° C, first of all to establish experimentally any deviations in the stress-creep-rate curve at low stresses for this material. In parallel with these experiments we have carried out creep tests to measure the amount of grain-boundary sliding in order to see if it is possible to explain the increasing stress-dependence of the creep-rate in terms of grain-boundary sliding.

2. Experimental Procedure

Two austenitic stainless steels of the 20 wt % Cr/35 wt % Ni type, designated A and B, have been examined in this work. The full chemical compositions are listed in table I. Steel B contains about 0.5 wt % Ti and Al, whereas no intentional additions of these elements had been made to steel A. Both alloys had been melted under high vacuum.

The two materials were used in a fine-grained version (grain-size 25 μm for both alloys) and a coarse-grained version (grain-size 120 μm for A and 100 μm for B). The 25 μm grain-size was produced by annealing steel A at 1000° C for 10 min and steel B at 1100° C for 5 min; the coarser grain-size was obtained by annealing alloy A at 1150° C for 1 h and alloy B at 1150° C for 15 min. The fine-grained condition of A was used for establishing a precise relation between stress and creep-rate, whereas the coarse-grained variety was only used for measurements of grain-boundary sliding. To ensure a similar microstructure as regards carbides in the two states of steel A, the coarse-grained material was annealed at 1000° C for 10 min after the 1150° C treatment. All anneals were followed by a quench in water.

The creep-testing was carried out in conventional single lever creep machines for constant load and equipped with double differential transformers for continuous recording of the creep-strain. All creep experiments were made at 700° C at loads between 1 and 25 kg/mm^2. The temperature was controlled within ± 0.5° C. In order to get a satisfactory reproducibility for the lowest creep-rates, i.e. down to about 2×10^{-5} %/h, the strain gauges were trimmed to a high sensitivity of about 2 μm and, furthermore, silica was used in the transmission pins from the specimen to the transformers to reduce disturbances due to temperature variations. It should be emphasised that special care was taken to assure

TABLE I Chemical composition of steels used (wt %).

Designation of alloy	C	Si	Mn	P	S	Cr	N	Ti	Al	N
A	0.008	0.13	0.52	0.007	0.009	19.7	34.8	0.01	0.006	< 0.003
B	0.015	0.45	0.42	0.007	0.010	19.7	34.1	0.42	0.62	0.014

equivalent microstructure in the specimens and precise creep conditions so as to obtain accurate and reproducible creep-rates. For instance, the specimens for each series were taken from the same bar of the alloys.

The methods adopted here for measuring grain-boundary sliding are based on measurements on the surface. The main technique employed was the one originally suggested by Brunner and Grant [13]. By means of transverse grid lines the components along the stress axis of the offsets due to grain-boundary sliding, u_t, are measured at every intersection between a chosen grid line and the boundaries. By averaging these offsets along a transverse grid line according to the following expression

$$\epsilon_{gb} = \frac{1}{t}\sum_{0}^{n}(u_t \tan\theta) \qquad (2)$$

the grain-boundary deformation is obtained [13]. Here t is the length of the transverse line, n is the number of transverse grain-boundaries, and θ is the angle between the stress axis and the grain-boundary. When the measurements according to this method had been completed, it was brought to our attention that a method based upon measurements of the transverse component (w) of the grain-boundary sliding along a longitudinal line [14, 15] has proved to give exactly the same amount of sliding as when measurements were made in the specimen interior by means of internal markers [15], The grain-boundary sliding experiments were therefore supplemented by two control measurements according to this latter technique. The deviation between the two methods was found to be very small.

Since the amount of grain-boundary sliding obtained for reasonable creep times necessarily decreased at the lower stress levels, we had to search for a method where smaller grain-boundary offsets could be measured. By plating the specimen with copper and sectioning it along a longitudinal surface, followed by a polish and light etch, the component of the grain-boundary sliding normal to the specimen surface, v_l, could be measured with good precision in an optical microscope. Here, we chose to measure along a line parallel to the stress axis. The strain due to grain-boundary sliding is given by an expression analogous to equation 2:

$$\epsilon_{gb} = \frac{K}{l}\sum_{0}^{n} v_l. \qquad (3)$$

K is a constant and l the length of the longitudinal line. K is extremely difficult to calculate from first principles. However, in the present case it can be determined experimentally by making two separate measurements, first applying the method according to equation 2 and then using the method according to equation 3. In this work we have found that K is very close to unity, which is in close agreement with earlier results by Graeme-Barber [15].

The creep specimens intended for the grain-boundary sliding measurements were machined with two flat and parallel surfaces, so as to make it possible to scribe grid lines on the specimen surface. The lines were produced by rubbing a cloth containing 6 μm diamond powder across the specimen. Since the machining operation introduces a thin cold-worked surface layer and since the sliding measurements are made directly on the surface, the final anneals at 1000° C were made after the machining for these specimens. In order to avoid oxidation and other contamination during the creep test, the specimens for these measurements were sealed in bellows of stainless steel. The bellows were evacuated and filled with helium or argon. The bellows were mechanically very weak and the load they absorbed was negligible. The creep tests were interrupted in the secondary stage at strains between 1.4% and 18%; the majority of the tests was interrupted below 8%. Each value of ϵ_{gb} was based on measurements of the offsets at about 200 grain-boundaries.

3. Results and Discussion

3.1. Stress/Creep-rate Relationship

Sherby and Burke [16] distinguish between three creep-ranges, the low, intermediate and high-stress creep-range for high-temperature creep, i.e. at temperatures above 0.5 T_m. Compared with the intermediate range the low-stress range is characterised by a weaker stress-dependence of the creep-rate and the high-stress range by a stronger stress-dependence. The creep process in the intermediate range is associated with dislocation climb and the stress-dependence obeys a power law, $\dot{\epsilon} = A\sigma^n$, where the exponent n for pure metals and solid solutions usually is between 4 and 5. The increasing stress-dependence in the high-stress region has been assigned, in two theories by Weertman [17] and Barrett and Nix [18], to the generation of a high vacancy concentration at high stresses. As already discussed in the introduction, the decreasing stress-dependence at low stresses is most commonly attributed to diffusion-creep.

For the fine-grained (25 μm) steel the three creep-ranges are clearly discernible (A, fig. 1). For the fine-grained variety of A we can identify the low stress creep-range below 2×10^{-3} %/h, the intermediate range between 2×10^{-3} %/h and 2%/h and the high-stress range above 2%/h. In accordance with the general observation in pure metals and solid solutions [16] we obtain a value of 4.75 for the exponent, n, in the power law for the intermediate creep-range for alloy A in both grain-sizes.

The Ti/Al-bearing alloy B precipitates finely-dispersed particles of γ' [$Ni_3(Ti, Al)$] during creep at 700° C. The size of the particles is about 150 Å. This has been demonstrated in a previous work [19]. Precipitation of γ' is rapid compared with the creep-testing times. From fig. 1 it is evident that this precipitation strengthens the alloy considerably. The stress-dependence of the creep-rate in the intermediate creep-range corresponds to an exponent of about 7.5 in the power law and is steeper than for alloy A (fig. 1). Such a behaviour is characteristic for materials hardened by a second phase [16, 20]. Both grain-size varieties of steel B exhibit decreasing stress-dependence of the creep-rate at low stresses, analogous to alloy A. The creep-rates in the low-stress range indicate a grain-size-dependence such that the creep is inversely proportional to a power of the grain-size slightly larger than 1. The grain-size-dependence of the onset of the low-stress creep supports the view that the loss of creep strength in this range is not due to coalescence of the γ' precipitates.

In fig. 1 there is also a line inserted corresponding to grain-boundary diffusion-creep according to equation 1 for 700° C and a 25 μm grain-size. For the grain-boundary diffusion term ωD_{gb} a recent experimental value of Smith and Gibbs [21] for an austenitic stainless steel has been used: $8.3 \times 10^{-7} \times (\exp -43400/RT)$, and for the geometrical constant B a value of $150/\pi$. We notice that the observed creep-rates fall below the diffusion creep line by more than an order of magnitude at low stresses, where diffusion creep might be expected to be governing. Furthermore, the theory of diffusion creep requires that the creep-rate is directly proportional to the stress. Experimentally we do not find this. In the low-stress range the creep-rate of steel A is proportional to a power of the stress of about 3 and for steel B to a power of about 2. The fact that the creep-rate in the low-stress range is approximately proportional to (grain-size)$^{-1}$ is also inconsistent with the operation of grain-boundary diffusion-creep, which would give a power of − 3. Hence, there seems to be very little justification from these observations to propose that the decreasing stress-dependence at low stresses in the present alloys is caused by diffusion-creep.

A consequence of the stronger stress-depen-

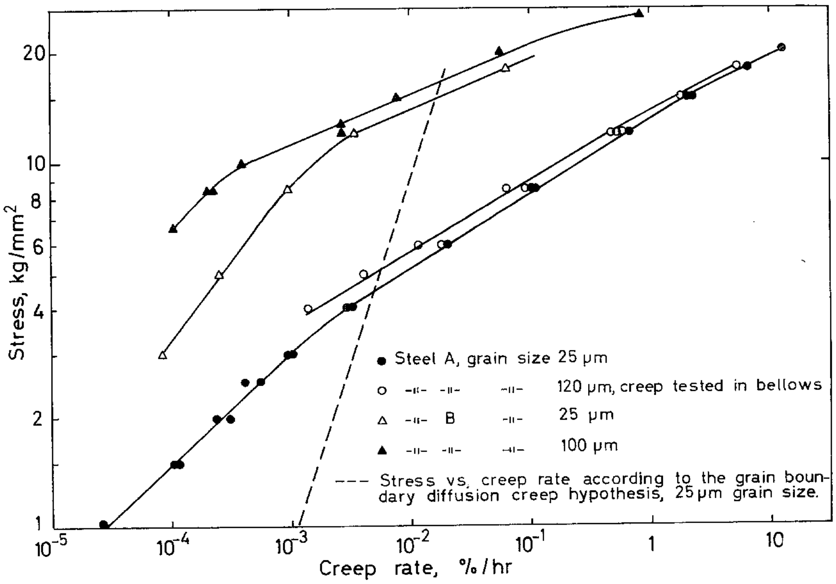

Figure 1 Stress versus creep-rate at 700° C for steel A and B.

dence of the creep-rate in the intermediate creep region for steel B and the weaker stress-dependence in the low-stress range (fig. 1) is that a precipitation-hardened alloy can be expected to display its highest creep strength relative to a solid solution with similar composition at the transition between the intermediate and the low-stress creep-range.

3.2. Grain-boundary Sliding and its Relation to Creep at Low Stresses

Measurements of grain-boundary sliding have only been made on steel A in the 120 μm grain-size variety. The reason why a larger grain-size was chosen for the sliding measurements than for the material for the creep-rate determinations, with which the sliding measurements should be correlated, was simply that it was found difficult to make precise measurements with the grid line technique for the smaller grain-size, 25 μm. $\dot{\epsilon}_g$Jb, i.e. the contribution of grain-boundary sliding to the total creep-rate, is simply calculated by dividing ϵ_{gb} by the creep time, thus assuming that ϵ_{gb} increases linearly with time. This seems to be a reasonable assumption since the total strain, ϵ, increases linearly from the very start of the creep test for the creep conditions employed here, and it has been shown that the ratio $\dot{\epsilon}_{gb}/\epsilon$ is approximately constant throughout the entire creep test [23]. To correlate the measurements of $\dot{\epsilon}_{gb}$ with the stress/creep-rate relation for the fine-grained material (fig. 1) the $\dot{\epsilon}_{gb}$ data have to be transformed to the 25 μm grain-size. Evaluation of data by Barrett *et al* [22] on copper indicates that $\dot{\epsilon}_{gb}$ is proportional to a power of the grain-size between -1 and $-1/2$. In fact recent work on the same material as steel A suggests that the power is -1 for the present alloy [19]. If we assume that the low-stress creep region is due to grain-boundary sliding, the results for the two grain-size versions of steel B in fig. 1 also suggest a power of about -1 (cf. section 3.1). The measured $\dot{\epsilon}_{gb}$ data have therefore been transformed to the 25 μm grain-size, assuming that $\dot{\epsilon}_{gb}$ is proportional to (grain size)$^{-1}$.

The ratio between ϵ_{gb} and the total creep-strain, ϵ, is approximately constant at high stresses, but increases with decreasing stress below a certain stress level as shown in fig. 2. Garofalo *et al* [24] have reported a similar stress-dependence of ϵ_{gb}/ϵ for a 18 wt % Cr/12 wt % Ni/Mo steel creep-tested at 704° C (fig. 2). As we can see from fig. 3, this behaviour implies that $\dot{\epsilon}_{gb}$ cannot be accounted for by a single power law for the stress range examined here.

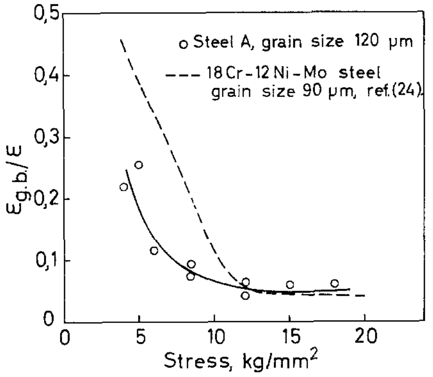

Figure 2 The ratio between the creep strain due to grain-boundary sliding, ϵ_{gb}, and the total strain, ϵ, versus creep stress for steel A at 700° C. The analogous curve for an 18 wt % Cr/12 wt % Ni/Mo steel at 704° C by Garofalo *et al* [24] is also inserted.

Fig. 3 shows the values of $\dot{\epsilon}_{gb}$ for steel A extrapolated to a grain-size of 25 μm, together with the creep-rate data for the same material at different stress levels. If the stress creep-rate relation for the low-stress region is extrapolated to higher stresses, the agreement with the corrected ϵ_{gb} values is very convincing. The agreement is good up to about 12 kg/mm^2 where the $\sigma - \dot{\epsilon}_{gb}$ curve deflects and takes a course roughly parallel to the $\sigma - \dot{\epsilon}$ curve. Apparently this break corresponds to the break in the (ϵ_{gb}/ϵ) versus σ relation (fig. 2). The coincidence between the low-stress creep curve and the $\dot{\epsilon}_{gb}$ data is a strong support that the low-stress creep-range and the decreasing stress-dependence of the creep-rate there is a result of the increasing contribution of grain-boundary sliding to the total strain at lower stresses. In view of the difficulties with the diffusion-creep hypothesis, as shown in the previous section, we suggest that the deflection in the stress/creep-rate curve at low stresses in the materials examined here is due to grain-boundary sliding. It should also be mentioned that the stress-sensitivity of the creep-rate in the low-stress range was in close agreement with the stress-dependence found for the rate of grain-boundary sliding in Mg [15]; the exponent in the power law was about 3 and 3.1 respectively.

4. Summary and Conclusions

(i) Careful determinations of the stress/creep-rate relation have been performed at 700° C for two

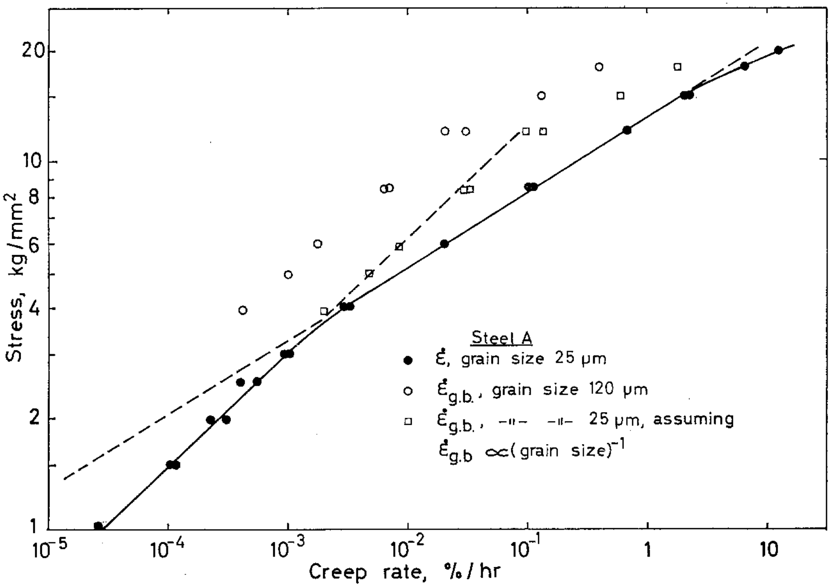

Figure 3 The creep-rate due to grain-boundary sliding, ϵ_{gb}, and the total creep-rate, ϵ, versus stress at 700° C for steel A.

20 wt % Cr/35 wt % Ni steels, A and B. A is a solid solution while B contains about 0.5 wt % Ti and Al and is strengthened by γ' that precipitates at 700° C. Both alloys display a low-stress creep-range in which the stress-dependence of the creep-rate is weaker than at higher stresses. For steel A with a 25 μm grain-size this implies that the creep-rate will be overestimated by about one order of magnitude at 1 kg/mm^2 by extrapolating creep data from higher stresses.

(ii) The stress-dependence of the creep-rate in the low-stress region can be described by a power law where the exponent for steel A is about 3 and for steel B about 2. These results contradict the hypothesis that the low-stress creep-range is caused by diffusion-creep, which requires a linear relation between creep-rate and stress. The measured creep-rates at low stresses are also below the rates predicted by the grain-boundary diffusion-creep theory by more than an order of magnitude.

(iii) Measurements of grain-boundary sliding have been carried out on steel A. It has been shown that the creep-rate caused by grain-boundary sliding correlates well with the stress/creep-rate relation at low stresses. Therefore we suggest that the low-stress creep-range, at least for the materials examined here, is a consequence of the increasing contribution of grain-boundary sliding to the total strain with decreasing stresses.

Acknowledgements

The authors thank AB Atomenergi for permission to publish this paper and Dr G. Östberg for valuable discussions and continuous support. Our thanks are further due to Mrs E. Brunberg and Mr R. Anglert for their able technical assistance.

References

1. J. E. HARRIS and R. B. JONES, *J. Nucl. Matls.* **10** (1963) 360.
2. J. E. HARRIS, R. B. JONES, and G. W. GREENWOOD, Central Electricity Generating Board, Berkeley Nuclear Laboratories, Internal Report RD/B/N. 172 (September 1963).
3. R. B. JONES, *Nature* **207** (1965) 70.
4. *Idem*, *J. Nucl. Matls.* **19** (1966) 204.
5. I. M. BERNSTEIN, *Trans. Met. Soc. AIME*, **239** (1967) 1518.
6. R. L. COBLE, *J. Appl. Phys.* **34** (1963) 1679.
7. R. L. SQUIRES, R. T. WEINER, and M. PHILLIPPS, *J. Nucl. Matls.* **8** (1963) 77.
8. I. P. BELL, J. STANDRING, P. C. L. PFEIL, G. H. BROOMFIELD, K. Q. BAGLEY, and A. S. FRAGER, 69th Annual Meeting Amer. Soc. for Testing Materials (June 1966).
9. R. B. JONES, Conf. on structural processes in high-temperature materials (AERE, Harwell, June 1967).
10. W. VICKERS and P. GREENFIELD, *J. Nucl. Matls.* **24** (1967) 249
11. L. E. RARATY, *ibid* **20** (1966) 344.
12. R. LAGNEBORG, *Met. Sci. J.* **1** (1967) 172.

13. H. BRUNNER and N. J. GRANT, *Trans. Met. Soc. AIME* **215** (1959) 48.
14. R. L. BELL, C. GRAEME-BARBER, and T. G. LANGDON, *ibid* **239** (1967) 1821.
15. C. GRAEME-BARBER, Ph.D. thesis (London University, 1967).
16. O. D. SHERBY and P. M. BURKE, *Prog. Materials Sci.* **13** (1967) 325.
17. J. WEERTMAN, *J. Appl. Phys.* **28** (1957) 362.
18. C. R. BARRETT and W. D. NIX, *Acta Met.* **13** (1965) 1247.
19. R. LAGNEBORG, *J. Materials Sci.* **3** (1968) 596.
20. *Idem*, to be published in *J. Materials Sci.*
21. A. F. SMITH and G. B. GIBBS, CEGB Report No. RD/B/N. 908.
22. C. R. BARRETT, J. L. LYTTON, and O. D. SHERBY, *Trans. Met. Soc. AIME* **239** (1967) 170.
23. F. GAROFALO, "Creep and Creep Rupture" (Macmillan, New York, 1965) p. 139.
24. F. GAROFALO, O. RICHMOND, W. F. DOMIS, and F. VON GEMMINGEN, *Proc. Inst. Mech. Eng.* **178** (1963-1964) 1/31.