Corrosion Science, Volume 35, Issues 5-8,
Advances in Corrosion and Protection, 1993, Pages 979-981, 983-987

THE INFLUENCE OF IMPOSED STRAIN RATE ON FRACTURE OF SURFACE OXIDES

by P. Hancock, J. R. Nicholls and K. Mahmood
School of Industrial and Manufacturing Science
Cranfield Institute of Technology,
Cranfield, Bedford, UK

SYNOPSIS

The mechanical properties of chromium rich scales formed on 304 stainless steel have been investigated as a function of deformation rate and operating temperature. At 900°C at slow strain rates < 10^{-6} per second no cracking was observed at strains up to 10%. At rapid strain rates in excess of 10^{-4} per second oxide cracking was found to be independent of strain rate and controlled by the fracture toughness of the oxide. In the intermediate region, with strain rates between 10^{-4} per second and 10^{-6} in the temperature range 700° to 950°C, the behaviour is determined by the creep deformation and fracture mode of the oxide. The mechanism of surface oxide failure is examined and an equation to predict cracking density over the full range of both monotonic tensile and creep fracture modes is suggested.

INTRODUCTION

Components operating at high temperatures, rely on the formation of protective surface oxides to prevent further oxidation in working environment. However, during operation most components are subject to applied stresses. These may be applied at high rates of strain, for example, in rapidly alternating loading or during rapid thermal cycling. Much lower strain rates would be generated during isothermal creep deformation. The alloys used for high temperature service are developed to have adequate strength, ductility and creep resistance to withstand these severe operating conditions but the protective surface oxides have more limited resistance to deformation yet to maintain their protective role they must not crack during service.

Failure by cracking of these surface oxides will lead to loss of protection and hence their mode of failure is particularly important. The strain rates that they are subjected to during exposure can vary by several orders of magnitude. At rapid rates of loading, the fracture behaviour of the scale would be expected to be determined by the fracture toughness of the protective scales. Measurements of the fracture toughness of the surface scales have previously been measured by two of the authors in the temperatures range 20° to 950°C under rapid loading[(1)]. Under creep loading it has been suggested that the effect of the applied creep rate

(ε) on surface oxide cracking can be described[2-5] by a power law of the form

$$d \alpha \frac{1}{(\varepsilon)^n} \quad \textbf{1}$$

where d is the average intercrack spacing between through thickness cracks in the oxide, ε is the applied strain rate and n is a strain rate exponent.

The mechanism of surface oxide failure is obviously dependent upon applied strain rate and temperature but how these variables might influence surface fracture over wide ranges of strain rate and temperature is considered to be worthy of further study.

EXPERIMENTAL

For this study it was necessary that the test pieces be manufactured from foil to ensure that a substantial fraction of the applied load would be carried by the oxide resulting in oxide fracture at some point in the test. 304 stainless steel, an alloy forming a predominantly Cr_2O_3 oxide was selected for compatibility with other oxide fracture studies undertaken by the authors[1]. The exact nature of the substrate is not critical as the study investigates through thickness crack formation and not spallation (interfacial crack formation).

The 304 stainless steel used in this study had a composition 18.26 Cr, 8.7 Ni, 0.3 Si, and 0.06 C was used throughout, in the form of 165μm thick foil.

The specimens were preoxidised in air for 100 hours at 900°C to ensure that the oxide thickness and composition was similar on all specimens. The thickness of the oxide was ~1.6μm and the composition of the oxide approximated to Cr_2O_3 with the presence of other contaminating sesquioxides such as $(Fe\ Cr)_2O_3$ and $(FeMn)_2O_3$ through the scale. The test temperatures ranged from 20 to 900°C with a comprehensive series of tests made at temperatures of 700°C, 800°C and 850°C and 900°C. Two types of tests were used, both were conducted in air:-

(i) Standard tensile tests were made on specimens produced to ASTM standard E-345-81. An Instron testing machine was used with five different cross head speeds ranging from 0.25mm per minute to 125mm per minute. As the specimen gauge length was 50mm it was possible to use strain rate of $8.33x10^{-5}$ per second up to $4.17x10^{-2}$ per second.

(ii) Creep tests were performed in a conventional beam loading machine modified to take the same size specimens as used in the tensile tests. Creep tests at 700°, 800°, 850° and 900°C were made at loads calculated to give strain rates between 10^{-5} and 10^{-8} per second. Extreme care had to be taken with both creep and tensile tests to ensure that no damage was done during set up and counterbalancing was used to avoid any inadvertent loading. All the specimens were deformed to 10% strain in order to ensure saturation cracking of the surface oxides.

After testing the specimens were furnace cooled and then examined by both optical and scanning electron microscopy. The crack spacing (d) on each specimen was measured at three different positions to obtain an average value. It is accepted that some growth in the length of cracks may have occurred during cooling, however, as the specimens were strained sufficient to result in a saturation of the crack density no new crack formation between existing cracks is to be expected.

A typical example of cracks formed during testing is shown in Figure 1.

RESULTS OF CRACK MEASUREMENTS

Values of the average crack spacing (d) were measured on both creep and tensile tests.

At low strain rates of $< 10^{-6}$ per second no oxide cracking was observed at 900°C, even after 10% strain. This is in agreement with similar findings by earlier workers[5,6,7] who also found no cracking at high temperatures and low strain rates. This effect has been attributed to the creep rate of the oxide being faster than the applied deformation rate imposed by the strain of the underlying metal. This work would support that hypothesis.

At creep rates between 10^{-6} and 10^{-4} per second cracking did occur and the results are shown in Figure 2. In this region the crack density is shown to depend upon both the applied strain rate and the applied temperature.

At the higher imposed strain rates which were obtained during tensile testing, also shown in Figure 2, it can be seen that the cracking density is dependent upon temperature but is independent of applied strain rate at strain rates greater than ~10^{-4} per second.

It seems reasonable to consider that, at the higher strain rates, the oxide failure will be dominated by fracture toughness considerations. In the creep region, creep deformation and a viscous sliding of oxide segments have been suggested to explain the effects observed[2-5]. Both mechanisms appear to be reasonable but their interdependence is not obvious. To examine the dependence of crack density on measurable material properties the two areas have been examined in greater detail.

Cracks produced during tensile deformation

The values of average crack spacing (d) found during tensile testing at different strain rates are shown on the right hand side of Figure 2. The scale used shows clearly the independence of strain rate effects but does not identify, in sufficient detail, the precise influence of temperature on d. This has been replotted in Figure 3 and shows how the crack separation d increases with increasing test temperature.

In the region where oxide failure is independent of strain rate it would be expected that oxide fracture would be related to fracture toughness. The critical

stress indensity factor K_c has been measured for surface oxides over the temperature range under investigation (20-900°C) and the results are reproduced in Figure 4[(1)]. By combining the data plotted in Figure 3, with that presented in Figure 4 at the relevant test temperatures, one may show that to a first approximation d^2 is proportional to K_c, Figure 5. Hence under rapid loading, such as exists during thermal shock or under a transient load cycle fracture of the scale is shown to depend upon the fracture toughness of the surface oxide.

Cracks produced during creep deformation

At the lower strain rates which are typically produced under creep loading the crack density is dependent upon both strain rate and temperature. Riedel[(2)] has developed an analysis for oxide fracture which, if considered in uniaxial tension, can be written in the simplified form

$$d = \left(\frac{8h\sigma_c}{\eta}\right)^{‰} \left(\frac{1}{\varepsilon}\right)^{‰} \quad \textbf{2}$$

where h is the oxide thickness, σ_c is its critical fracture stress and η is a viscosity coefficient related to the oxide metal interfacial layer.

For an oxide of constant thickness such as used in this investigation, at a fixed temperature, the first term becomes a constant and hence the equation can be rewritten as:-

$$d = \left(\frac{A}{\varepsilon}\right)^{‰} \quad \textbf{3}$$

where A is a constant for any given temperature and film thickness.

Later work by Schutze[4,5] on a range of steels and superalloys demonstrated that the exponent was generally less than 0.5 and values of between 0.26 and 0.44, depending upon the alloy under test, were found to give a better fit to his experimental data. However, the exponent value of 0.5 has theoretical significance in terms of viscous sliding or creep. Further, if it is intended to consider both fast and slow strain rates then equations (2) and (3) both suggest that as the strain rate increases, d should approach zero. But, it was shown in the section on tensile testing, that as the strain rate increases, the value of d assumes a characteristic spacing, which is independent of strain rate, controlled by the fracture toughness of the oxide.

Hence it would seem more appropriate to rewrite the Riedel equation as

$$d^2 = \frac{A}{\varepsilon} + C \qquad \textbf{4}$$

where C is a measure of the crack spacing produced at high strain rate.

Using the tensile data results at strain rates greater than 10^{-4} per second, it has been shown that d reaches a constant value and that d^2 is directly proportional to K_c the critical stress intensity factor of the oxide. To check whether this also applies to the creep data, the creep data has been replotted as d^2 vs $1/\varepsilon$ in Figure 6. This figure shows that the data does give a reasonable fit to equation (4) and the increasing slopes of the lines with increasing temperature show that A is

temperature dependent as would be expected if some form of viscous flow is a controlling factor.

The intercept on Figure 6 when $1/\varepsilon \rightarrow 0$ i.e. $\varepsilon \rightarrow \infty$ means that this d^2 should be a limiting value. In the section on tensile deformation this was shown to be proportional to K_c the critical stress intensity factor.

To explore whether the creep results would predict the same effect, a plot of the intercept on Figure 6 at $1/\varepsilon = 0$ should give a value of d^2 (at $\varepsilon \rightarrow \infty$) which should be directly proportional to the critical stress intensity factor. Figure 7 plots the intercept C against K_c and it is seen to be a very reasonable linear relationship. It must be emphasised that the oxide scale tested was approximately of constant thickness, resulting from the 100h preoxidation treatment at 900°C. Subsequent creep test at temperatures in the range 700-900°C resulted in only a limited further growth of this oxide. Hence this test procedure is unique in that the fracture toughness (K_c) of the scale can be changed (via test temperature) for a constant scale morphology and thickness (determined by the preoxidation treatment used).

This enables equation (4) to be reformulated in terms of K_c as:-

$$d^2 = \frac{A}{\varepsilon} + B\ K_c \qquad \textbf{5}$$

where the constant B is temperature independent.

This equation can now be used to represent all the data generated both in the tensile tests and in the creep tests. It shows the dependence of the crack spacing on the fracture toughness of the oxide and it enables the surface oxide fracture under both rapid loading, as in thermal cycling, and slow strain rate loading, as in creep deformation, to be reconciled. At high strain rates, the first term A/ε becomes insignificant, fracture behaviour is strain rate independent, and d is determined by the fracture toughness of the oxide. At low strain rates the first term is dominant but, as the applied strain rate is increased, there is always a characteristic minimum crack spacing value which is determined by the second term BK_c and this agrees with experimental observations.

CONCLUSIONS

1. At very low strain rates (smaller than 10^{-6} per second) and at high temperatures (greater than 800°C) there is no evidence of surface oxide cracking on 304 stainless steel even at strains as high as 10%. It is concluded that in this region the creep strain in the oxide can accommodate the superimposed deformation rates.

2. Under high strain rates (greater than 10^{-4} per second) imposed by rapid deformation or by thermal cycling. Fracture of the protective surface oxide occurs and the crack density is independent of strain rate. The crack density is however, temperature dependent and is determined by K_c the critical stress intensity factor of the surface oxide at the operating temperature.

3. In the intermediate creep region, crack density depends upon both temperature and strain rate. A simple single equation of the form:-

$$d^2 = \frac{A}{\varepsilon} + BK_c$$

can be used to describe both the creep and high strain rate oxide fracture, where A and B are constants. A is temperature dependent and B temperature independent. This equation has been shown to give a good approximation to the surface oxide cracking behaviour on 304 stainless steel.

REFERENCES

1. P. Hancock and J. R. Nicholls, Materials Science and Technology 4 398-406, (1988).
2. H. Riedel, Metal Science 16 569-574 (1982).
3. J. C. Grosskreutz, J. Electrochem Soc. 116, 1232-1237 (1969).
4. M. Schutze, Oxid. Met. 24, 199-232 (1985).
5. M. Schutze, Oxid. Met. 25, 409-422 (1986).
6. L. Berchtold, H. G. Sockel and B. Ilschner, Behaviour of High Temperature Alloys in Aggressive Environments 927-941, (1980). The Metals Society, London.
7. L. Berchtold, M. Welker, H. G. Sockel, Werkstoffe und Korrosion 37, 3, 145-149, (1986).

List of Figure Captions

Figure 1 Cracks formed on 304 stainless steel (preoxidised at 900°C) after testing at 850°C at a strain rate of 3.33×10^{-4} per second.

Figure 2 Distance between cracks in the surface oxide (d) as a function of temperature and strain rate.

Figure 3 Characteristic crack separation d as a function of temperature in the strain rate independent region.

Figure 4 The influence of temperature on the critical stress intensity factor K_c for surface oxides. (1)

Figure 5 Relationship between d^2 and the critical stress intensity factor K_c for the surface scale.

Figure 6 Relationship between d^2 and $1/\varepsilon$ derived under creep testing at various temperatures.

Figure 7 Intercept C from Figure 6 plotted against K_c the oxide fracture toughness

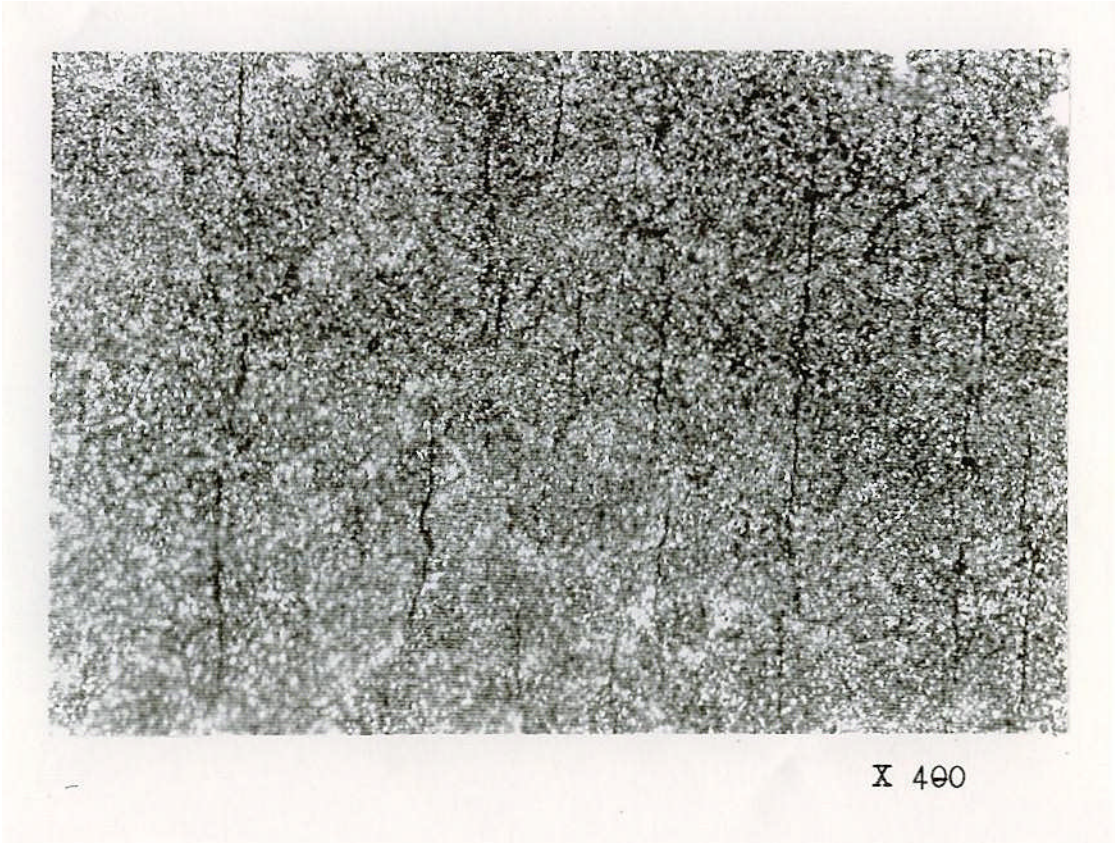


Figure 1 Cracks formed on 304 stainless steel (preoxidised at 900°C) after testing at 850°C at a strain rate of 3.33×10^{-4} per second.

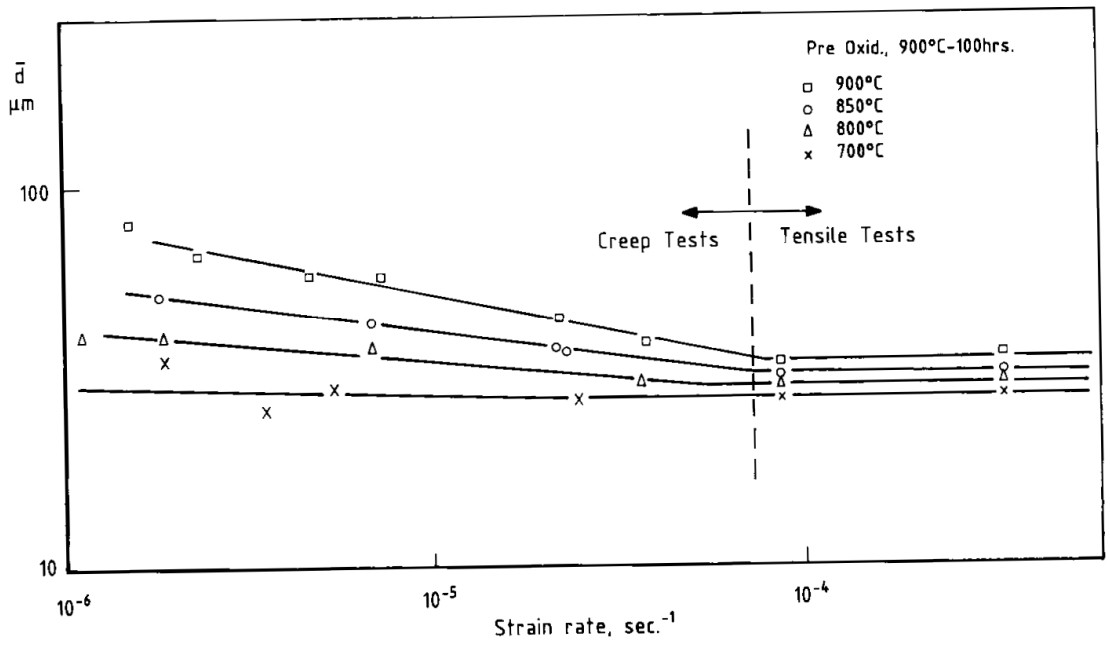


Figure 2 Distance between cracks in the surface oxide (d) as a function of temperature and strain rate.

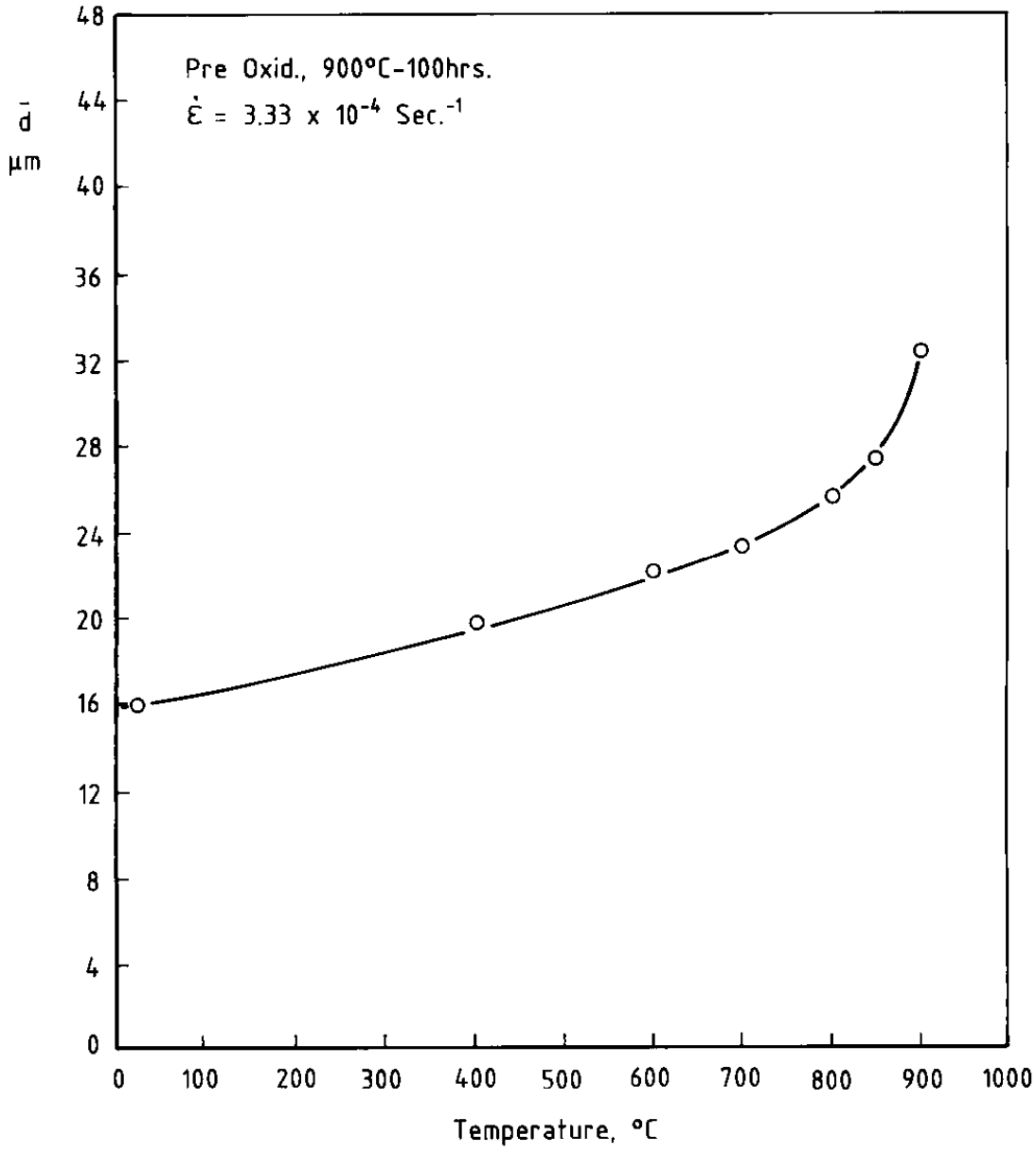


Figure 3 Characteristic crack separation d as a function of temperature in the strain rate independent region.

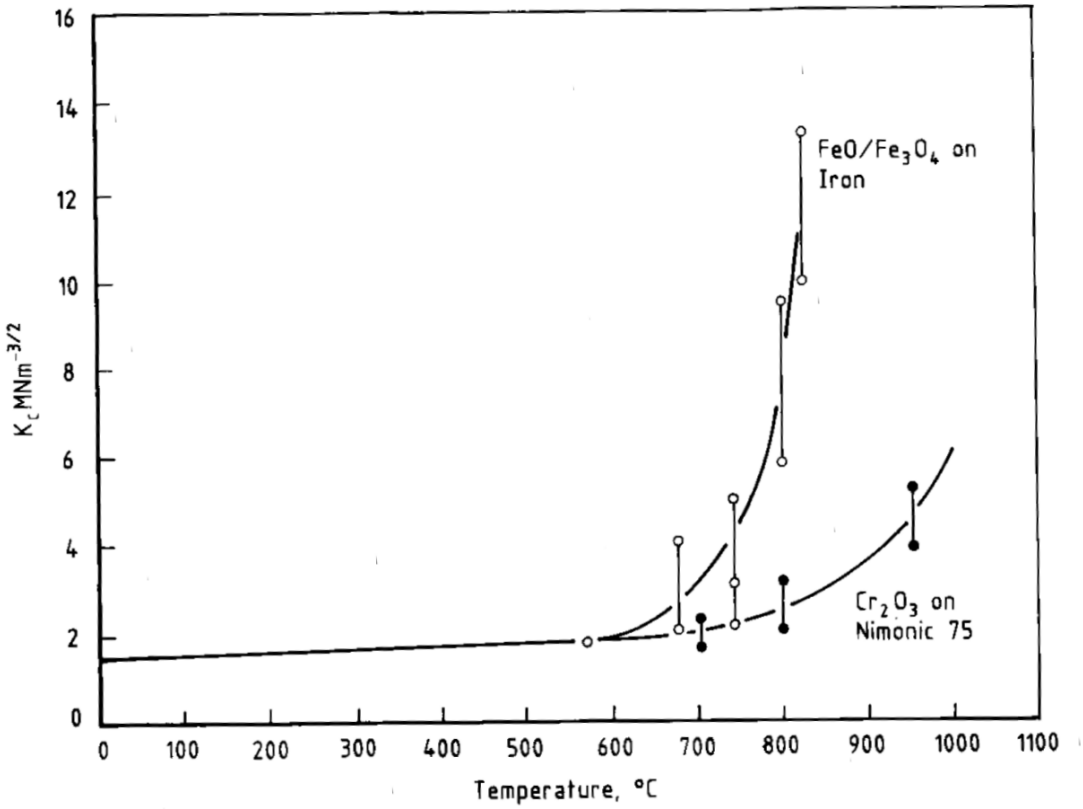


Figure 4 The influence of temperature on the critical stress intensity factor K_c for surface oxides. (1)

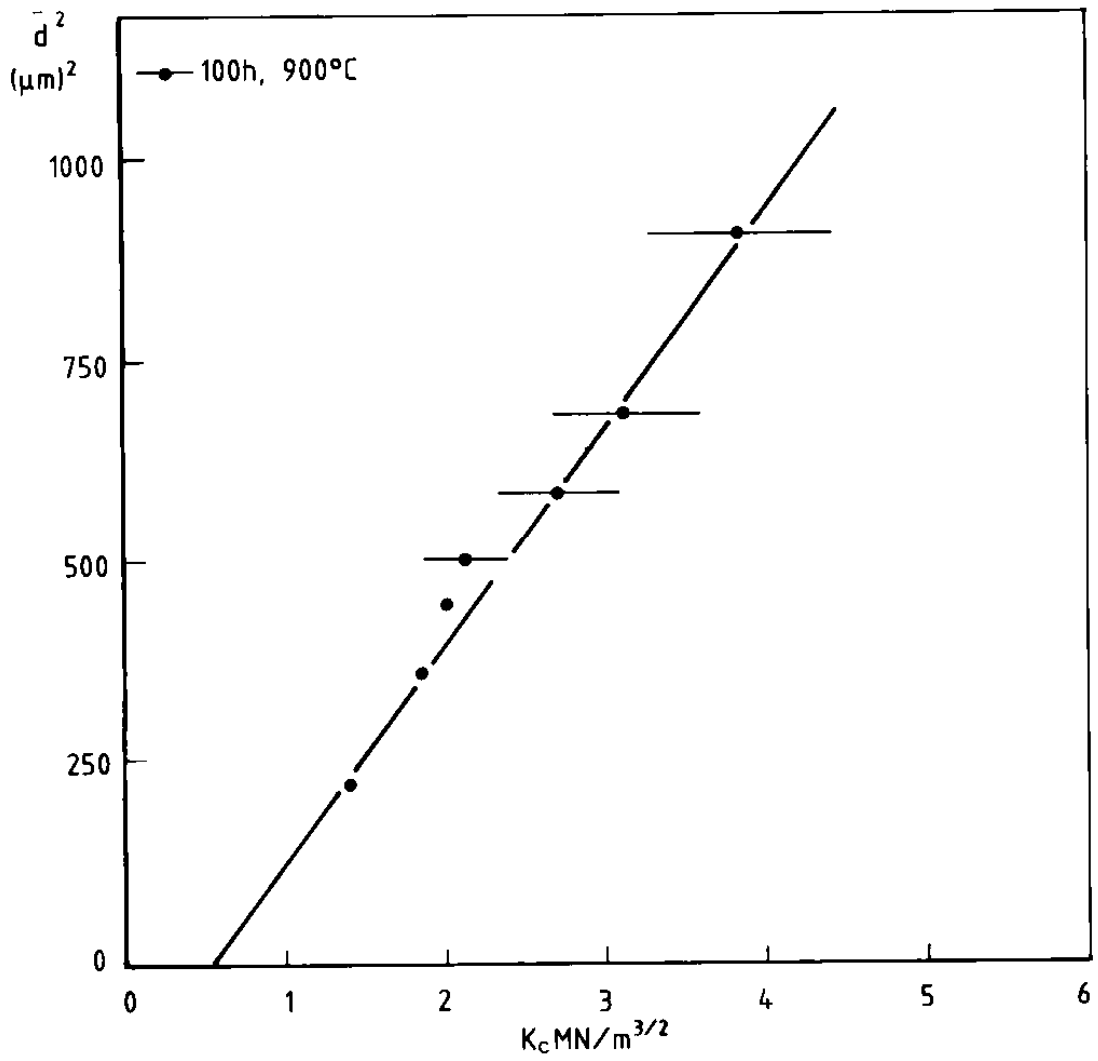


Figure 5 Relationship between d^2 and the critical stress intensity factor K_c for the surface scale.

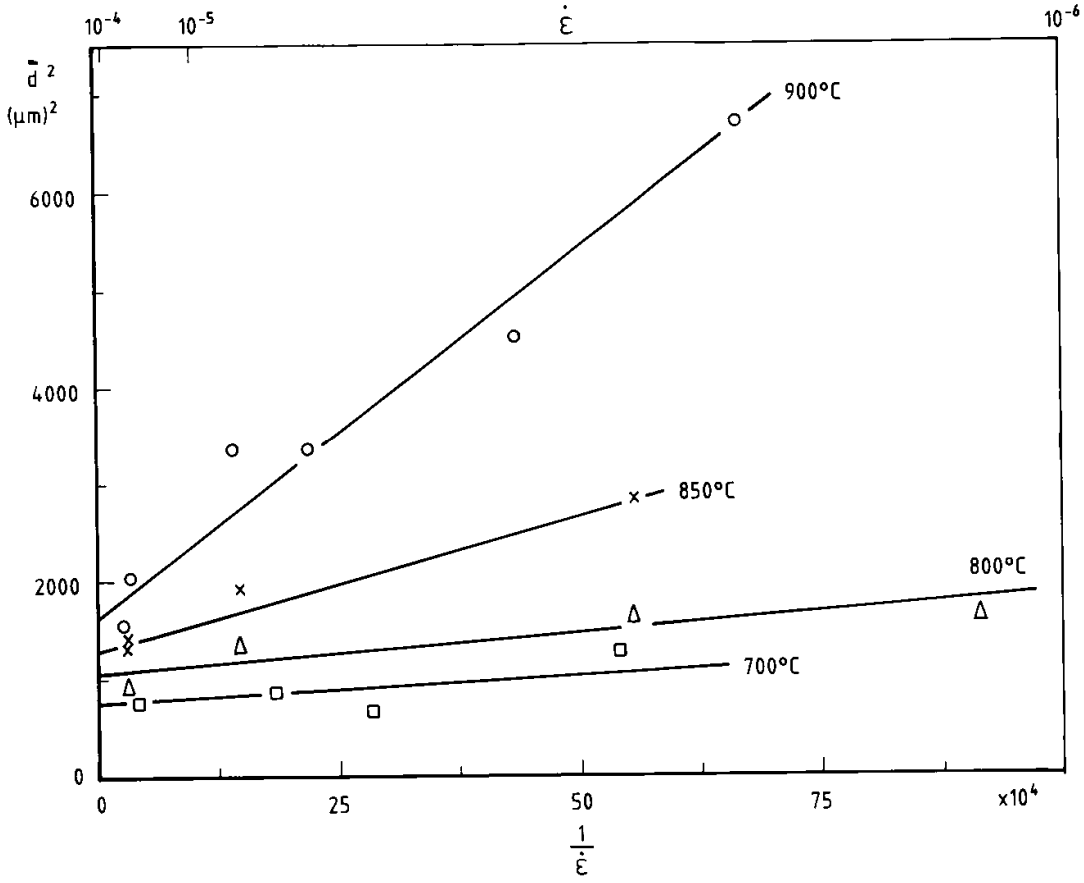


Figure 6 Relationship between d^2 and $1/\varepsilon$ derived under creep testing at various temperatures.

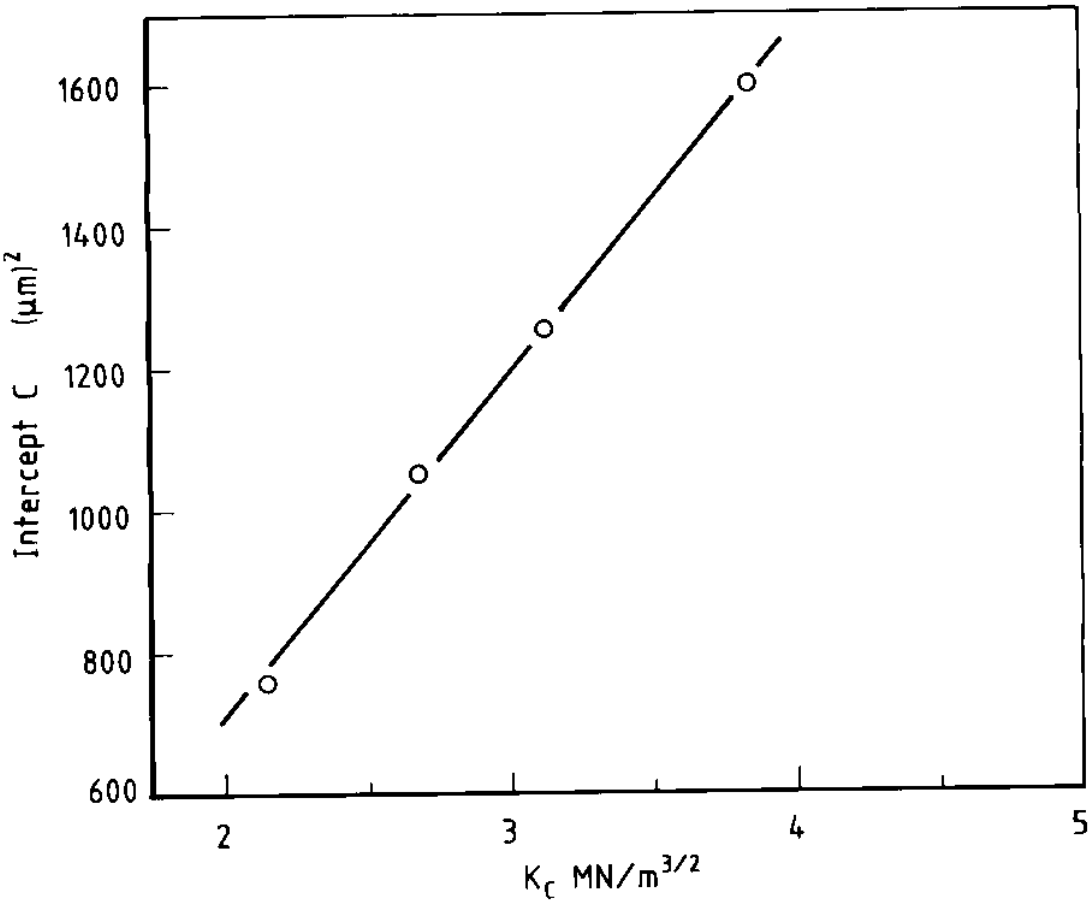


Figure 7 Intercept C from Figure 6 plotted against K_c the oxide fracture toughness



https://dspace.org/

The influence of imposed strain rate on fracture of surface oxides.

Hancock, P.; Nicholls, John R.; Mahmood, K.

1993-01-01T00:00:00Z
Elsevier Science B.V., Amsterdam.
http://dx.doi.org/10.1016/0010-938X(93)90316-9
P. Hancock, J.R. Nicholls, K. Mahmood, The influence of imposed strain rate on fracture of surface oxides, Corrosion Science, Volume 35, Issues 5-8, Advances in Corrosion and Protection, 1993, Pages 979-981, 983-987

Downloaded from a DSpace repository. https://dspace.org/