THE EFFECT OF TEMPERATURE, STRAIN RATE AND ENVIRONMENT ON THE DUCTILITY OF 304 STAINLESS STEEL

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The influence of strain rate and temperature (50–250°C) on the mechanical properties of 304 stainless steel has been investigated in both argon and liquid rubidium environments. A loss of ductility has been observed in argon when temperature or strain rate were increased, but not in liquid rubidium. Several explanations for this phenomenon have been considered, and mechanical heating during deformation in argon is favoured.

INTRODUCTION

It is widely known that environmental factors can significantly affect the mechanical properties of materials. Examples of detrimental environmental effects include metal induced embrittlement, stress corrosion cracking, hydrogen embrittlement and the Re binder effects. A number of workers (including Joffe (1), and Kramer and Demer (2)), have reported increases in mechanical properties which have been attributed to the influence of the environment in which the materials are strained; this effect is usually associated with the removal of surface defects or hard surface layers, resulting in a reduction in flow stress. In most of the above examples the change in mechanical properties can be attributed to a surface interaction rather than a bulk chemical change in the deforming solid.

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When screening a number of commercial alloys for possible embrittlement by liquid rubidium Trevena and Nicholas (3) have reported a surprising increase in the tensile ductility of several austenitic stainless steels (304, 321, 316, 347) and nickel based alloys (Nimonic 105, Nimonic 80A) when strained in contact with liquid rubidium. It was found that 304 stainless steel had a failure strain of 58% in argon which increased to 76% in liquid rubidium, when strained at 50 mm/min at 50°C. Pre-exposure to rubidium with subsequent mechanical testing in argon produced no increase in ductility. Unlike any of the environmental effects mentioned above the enhanced ductility in rubidium is associated with an increase in the rate of work hardening.

The object of this present investigation has been to determine what influence experimental conditions have on this ductility enhancement, and attempt to understand the causes of the effect. Both temperature and strain rate have been systematically varied during tensile tests in argon and liquid rubidium environments.

**EXPERIMENTAL MATERIALS AND TECHNIQUES**

The 304S15 stainless steel used in this study was obtained from Barpoint Ltd. in the form of 10 mm drawn bars. The composition is given in Table 1. Rubidium was supplied by Koch-Light Ltd. in 5 g ampoules with a nominal purity of 99.98%. 3 mm diameter tensile specimens were machined from the steel in the as-received condition (Figure 1) and tensile tests were conducted in both liquid rubidium and argon (control) environments, followed by detailed fractography of the failed specimens.

**TABLE 1 - Composition of 304 stainless steel**

<table>
<thead>
<tr>
<th>%</th>
<th>Cr</th>
<th>Ni</th>
<th>Si</th>
<th>P</th>
<th>Ti</th>
<th>Co</th>
<th>Cu</th>
<th>Nb</th>
<th>Mo</th>
<th>S</th>
<th>Mn</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>18.1</td>
<td>10.3</td>
<td>0.51</td>
<td>0.03</td>
<td>&lt;0.01</td>
<td>0.16</td>
<td>0.15</td>
<td>&lt;0.005</td>
<td>0.27</td>
<td>&lt;0.01</td>
<td>1.53</td>
<td>69.0</td>
</tr>
</tbody>
</table>

Two major pieces of apparatus were used for the experimental work; a high integrity glove box and an Instron 1195 tensile testing machine. The quality of the argon atmosphere within the stainless steel glove box was maintained by recirculation through a
purification train where molecular sieve and BTS catalyst remove both water vapour and oxygen, typical working levels of these impurities were 1 ppm.

Machined specimens were degreased in acetone, and tensile testing was carried out inside capsules machined from 321 stainless steel. Control specimens were loaded into a test capsule (Figure 2) which was then sealed, evacuated and back-filled with argon. When the capsule was loaded, it was attached to the Instron 1195 testing machine, heated to the desired experimental temperature and strained to failure at 50, 1 or .05 mm/min. These speeds correspond to strain rates of $5.5 \times 10^{-2}$ s$^{-1}$, $1.1 \times 10^{-3}$ s$^{-1}$ and $5.5 \times 10^{-5}$ s$^{-1}$, which we shall refer to as fast, intermediate and slow. Specimens to be tested in rubidium were loaded into an identical test capsule within the argon filled glove box, which was then filled with 15 cc of rubidium, sealed, and removed from the glove box. Specimens in rubidium were then tested in a similar manner to the control experiments. During the experiments the temperature was measured by a sheathed chromel-alumel thermocouple which was attached to the side of the test specimen. Separate capsules were used for argon and rubidium tests to avoid cross contamination.

Values of ultimate tensile stress (UTS) and elongation to failure ($\varepsilon_f$) were calculated from the load-displacement graphs and the specimen dimensions. Following the mechanical testing a selected number of specimens were examined for any evidence of chemical interaction.

**EXPERIMENTAL RESULTS**

Tensile data for all the experiments are summarised in Figure 3. The control (argon) data show a decrease in ductility over the range 50-250°C and it is interesting to note that as the strain rate increases so the low temperature ductility decreases.

The dramatic effect of rubidium on tensile properties can be seen in Figures 3 and 4. At 50°C both strength and ductility were higher in rubidium than in argon; as the test temperature increased to 250°C this effect was less noticeable. Note that there is little or no effect on macroscopic yielding, rather the rate of work hardening in rubidium is higher than in argon, thus necking is suppressed leading to increases in ductility and failure stress. In contrast to the control data the ductility of the steel in rubidium was NOT affected by the strain rate, so fast tests produced the largest
examples of enhancement.

Fractographic examination by SEM revealed that tests in both argon and rubidium produced ductile flow and dimpling, indicating that failure occurred by microvoid coalescence. No evidence was found of any significant difference in appearance between the rubidium and argon fracture surfaces. Optical metallography and electron probe micro-analysis was carried out on polished cross-sections of specimens which had been strained to failure and again no difference was found between argon and rubidium tests; no evidence was found of any chemical interaction between the steel and rubidium.

DISCUSSION

Before attempting to understand the influence of a liquid rubidium environment it is necessary to examine carefully the control data of the 304 stainless steel. At temperatures below 250°C in argon an increase in strain rate results in reduced ductility. Similarly, as the test temperature increases so the ductility decreases. So the highest ductility in argon is found at 50°C at the slow strain rate; increases in test temperature or strain rate reduce the ductility. However, when the steel is tested in rubidium at 50°C strain rate appears to have little effect on ductility. The larger ductility observed in liquid rubidium at fast strain rates is due to a decrease in the ductility in argon under these conditions, rather than an enhancement in rubidium.

The observed influence of temperature and strain rate, together with the available diffusion data, argue against a diffusion controlled mechanism for ductility retention in rubidium, which would be favoured by higher temperature and lower strain rates. EPMA studies of the failed specimens show no evidence of chemical interaction or interdiffusion between the steel and rubidium, thus suggesting that the change in flow characteristics does not occur as a result of changes in the bulk chemistry of the steel.

In attempting to explain the higher ductility in rubidium the authors have considered whether, by implication, the flow characteristics have altered as a result of surface interactions. The role of the surface during deformation has been studied, in metal single crystals and ceramics, where Joffe, and Kramer and Demer, have observed enhancements in mechanical properties which have been attributed to environmental
interactions (1,2). An explanation for the observed retention of ductility in rubidium is to consider whether changes could be induced in surface and near surface plasticity, either by an interaction between rubidium and the surface oxide or induced by adsorption. This model presupposes that changes in the near surface dislocation structure (activation of an increased number of surface sources, and the subsequent surface hardening), could have long range effects on the generation, mobility and interaction of dislocations within a polycrystal. However this mechanism for enhancement of ductility would not be in agreement with the conventional view of the deformation of polycrystals, where internal grain boundaries are presumed to dominate plastic behaviour.

If there is no evidence for a bulk chemical change in the steel which could explain the influence of rubidium on ductility, and the argument for a surface change dominating bulk plasticity is unproven, then what else could explain this phenomenon? The authors have considered whether the observations are an experimental artefact. Examination of failed specimens has confirmed the values of ductility which were measured in rubidium, so the liquid metal does not directly introduce an error. We have already concluded that the observed effect of faster strain rates at 50°C is to reduce the ductility of the steel in argon. At the slow strain rate in argon an increase in temperature results in a decrease in ductility. Could the faster strain rate cause internal heating of the specimen so reducing the ductility? This would explain the mechanical behaviour of the steel in argon. The presence of rubidium prevents the ductility loss at high strain rates; this suggests the possibility of the liquid metal acting as an efficient heat transfer medium, thus preventing the heating of the specimen. Liquid metals are used as coolants in the nuclear industry because of their highly efficient heat transfer characteristics; the thermal conductivity of rubidium is an order of magnitude greater than that of argon. The mechanical data could be explained if tests in liquid rubidium are isothermal, while tests in argon are heated by deformation. When tests were carried out in rubidium a temperature rise of less than 5°C was recorded by the thermocouple, whereas an increase of 10-15°C was occasionally detected in argon. This would not seem to be a large enough difference to account for the observed mechanical data, but accurate temperature measurement during a dynamic experiment is difficult because the thermocouple is not in intimate contact with the tensile specimen. However, it is possible to calculate the maximum temperature rise.
during deformation by making the following assumptions;
(i) that all of the mechanical energy produces heat,
(ii) there are no heat losses from the specimen, and
(iii) there is no temperature gradient within the gauge
length of the specimen. The maximum temperature rise
can then be calculated from the equation

\[ \frac{dT}{\Delta T} = \frac{dQ}{m \cdot c} \]  

(1)

where
- \( dT \) = rise in temperature
- \( dQ \) = energy input
- \( m \) = mass of gauge length
- \( c \) = specific heat capacity

substituting the following values in the equation

\[ dQ = 30 \ J \]
\[ m = 8 \times 10^{-4} \ kg \]
\[ c = 400 \ J \cdot kg^{-1} \cdot K^{-1} \]

which gives a value of greater than 90K for the
temperature increase.

This rise in temperature would be sufficient to induce a
loss of ductility in the steel.

Trevena and Nicholas (3) have previously reported a
higher ductility in rubidium than argon at high strain
rate for several stainless steels and nickel based
alloys; if the above explanation of the mechanical data
is correct then it will be necessary to demonstrate that
these materials show a loss of ductility with increasing
temperature. Further studies are in hand to
characterise the effect of temperature on mechanical
properties, to accurately measure temperature, and to
investigate the effect of other liquid environments.

CONCLUSIONS

1. Rubidium has not been found to embrittle 304SS,
between 50 and 250°C, at fast, intermediate and slow
strain rates.

2. At the slow strain rate the effect of increasing
temperature is to reduce the ductility of the steel.

3. In argon the effect of increasing strain rate is to
reduce the ductility of the steel.

4. In rubidium the ductility of the steel is
independent of strain rate.

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5. There is no evidence of any chemical interaction between the steel and liquid rubidium.

6. Heating during mechanical deformation could explain the effect of strain rate on ductility. In the absence of evidence for any physical interaction between the liquid metal and the solid, the possible cooling of the steel during deformation could explain the higher ductility in rubidium.

LIST OF REFERENCES


Figure 1  Geometry of tensile testpiece.

Figure 2  Schematic diagram of mechanical test capsule.
Figure 3

Figure 4