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Further studies of the effect of rubidium on the mechanical properties of an austenitic steel

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The effect of strain rate and temperature on the mechanical properties in argon and rubidium have been studied for a Type 304 austenitic steel. It has been shown that samples rapidly strained can experience major temperature rises in argon but not in rubidium. When sample temperatures are equal, there is no marked effect of the environment on mechanical behaviour. For both environments, the ductility and the strength of the steel decrease markedly as the temperature is raised from 50 to 150° C but are little changed by a further temperature rise to 250° C.

1. Introduction

It is widely known that changing an environment can affect the mechanical properties of materials. For example, a metal that is strong and ductile when stressed in air or an inert gas can fail prematurely by cleavage when stressed in a liquid metal environment [1–3]. Other important mechanical degradation processes include hydrogen embrittlement and stress corrosion cracking, to both of which liquid metal embrittlement can be compared [4, 5].

The chances and consequences of liquid metal embrittlement occurring when stressed components performing some critical role come into deliberate or accidental contact with a molten metal should be considered by prudent design engineers. When the possibility of using gas bottles for the long-term storage of the fission product Kr^{85} was being explored, the possibility of liquid metal embrittlement was identified since the gas decays to form rubidium, a low melting point alkali metal. Harwell workers [6] conducted tensile tests to assess the susceptibility of a number of commercial alloys to rubidium-induced embrittlement, and observed a surprising *increase* in the ductility — measured by the elongation to fracture, E_f — of a number of austenitic steels and face-centred cubic nickel alloys, as illustrated in Fig. 1. These tests had been conducted at the low temperature of 50° C, only 11° C above the melting point of rubidium, and the fast strain rate of 5.5×10^{-2} sec^{-1} to maximize the likelihood and severity of any liquid-metal embrittlement. The unexpected observation of enhanced ductility was accompanied by small but significant increases in the ultimate tensile strength (UTS) and the rate of work-hardening of the steels.

Why rubidium environments were beneficial was puzzling. X-ray diffraction analyses indicated that more transformation of the metastable austenitic steels to martensite had occurred during their deformation in rubidium environments, but this only complicated the puzzle. Chemical surveys using electron probe microanalysis did not reveal any evidence of rubidium diffusion into the bulks of steel samples, nor should any have been expected because of the short contact times, only a few minutes, and low contact temperatures. Published chemical stability data suggest the rubidium could have stripped the oxide films from the steel surfaces, and while this should have affected dislocation behaviour in the surface region it is implausible for it to have any significant effects on the bulk mechanical behaviour of the polycrystalline commercial alloys. The arrangement of the bottle material screening programme did not permit any more detailed investigation of the ductility enhancement; it was sufficient for the designers that embrittlement had not occurred. The experimental observations therefore remained an unresolved puzzle until an opportunity arose recently to reinvestigate the behaviour of the Type 304 austenitic steel. To maximize the relevance of this additional work, care was taken to use the same batch of steel and the same testing procedures as employed in the screening programme.

2. Materials and techniques

The rubidium used in this work was 99.98% pure material obtained as 15 g ampoules from Koch–Light Ltd., while the steel was the austenitic BS 304 S15 whose composition is given in Table I. The steel was bought from Barpoint Ltd as 10 mm diameter drawn bars, with a hardness of 190 VHN, from which tensile test samples of the form shown in Fig. 2 were machined. The surface of the gauge length section of the test pieces never had an average roughness, R_a, greater than 0.2 μm.

Each sample was degreased by ultrasonic agitation in acetone and posted into a high integrity glove box filled with recirculating argon purified to oxygen and moisture levels of 1 v.p.m. or better. Also posted was

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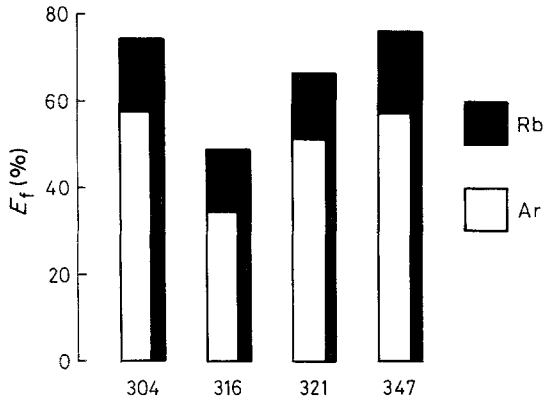


Figure 1 The elongation to fracture (E_f) values of some steels and nickel alloys tensile tested in argon and rubidium at 50°C using a strain rate of 5.5×10^{-2} sec^{-1}.

an AISI 321 stainless steel capsule (sketched in Fig. 3) in which the mechanical test was to be performed. One end of the sample was gripped by a bayonet device inside the bucket attached to the bottom of the capsule, and the other end was attached to the pull-rod that passed through the bellows in the top flange of the capsule. Rubber O-rings were used to make seals below the bellows and the pull-rod. The additional load due to the spring constant of the bellows, 1 N mm^{-2}, was insignificant compared to the applied loads of several kiloNewtons required to break the samples. If a test was to be conducted in rubidium, an ampoule was broken inside the glove box and its contents melted before being syringed into the bucket. The capsule was then sealed and posted out of the box.

The loaded capsule was attached to an Instron 1195 machine and a muffle furnace was fitted around the capsule bottom. The capsule was evacuated and fresh high-purity argon was introduced to produce a pressure selected to ensure that one atmosphere was approached but not exceeded at the test temperature. A water-cooled block was fitted to protect the O-ring seals of the capsule flanges and power supplied to the muffle furnace. The capsule temperature was measured by a stainless steel sheathed alumel–chromel thermocouple placed in the bucket, and was allowed to stabilize at or within 2° C of the selected experimental temperature – 50, 100, 150 or 250° C – for 5 min before the sample loading and extension was started. Three constant rates of extension were used in many tests: 0.05, 1 and 50 mm min^{-1}, corresponding to strain rates of 5.5×10^{-5}, 1.1×10^{-3} and 5.5×10^{-2} sec^{-1}, respectively. In a few tests, a very rapid extension rate of 500 mm min^{-1} was used which corresponded to a strain rate of 0.55 sec^{-1}. In some tests conducted at 50° C, attempts were made to measure the sample temperatures as will be described later. Load–extension curves were generated by the Instron 1195 for all the tests and used to estimate E_f and UTS values.

After the samples had been strained to failure, the experiment was ended and the capsule was reposted into the glove box for dismantling. The fracture surfaces of the samples tested in rubidium were cleaned by being immersed briefly in water and rinsed in ethyl alcohol. Subsequently, the fracture surfaces of most test pieces were examined using scanning electron microscopy.

TABLE I Composition of BS 304 S15 steel (wt %)

18.1 Cr, 9.0 Ni, 0.9 Mn, 0.59 Si, 0.48 Mo, 0.24 Co, 0.028 C, 0.027 P, 0.013 N, 0.012 S, 0.01 Ti, balance Fe.

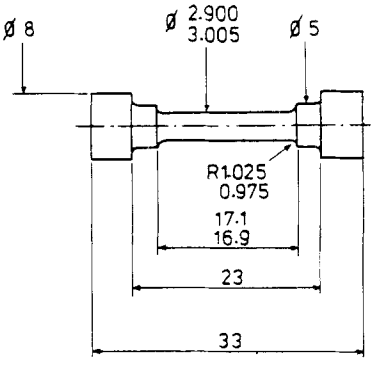


Figure 2 Tensile test sample (dimensions in mm).

3. Results and discussion

Three series of experiments were conducted to examine the effect of varying the test temperature and the strain rate, and to measure sample temperatures.

3.1. The effect of test temperature

Using a strain rate of 5.5×10^{-2} sec^{-1}, samples were stressed to destruction in argon and rubidium at test temperatures of 50, 100, 150 and 250° C. The effects of varying the test temperature are shown in Fig. 4. These and other mechanical property data are

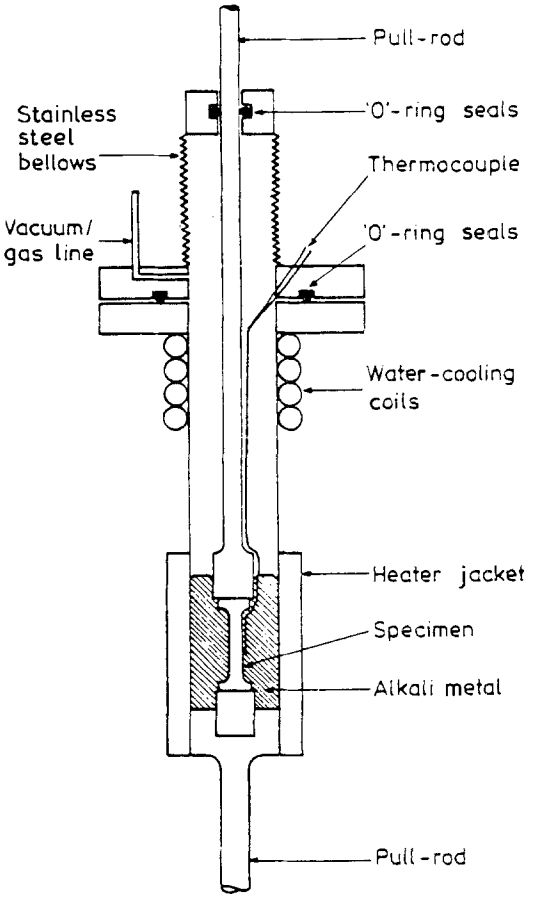


Figure 3 Schematic diagram of the tensile test capsule.

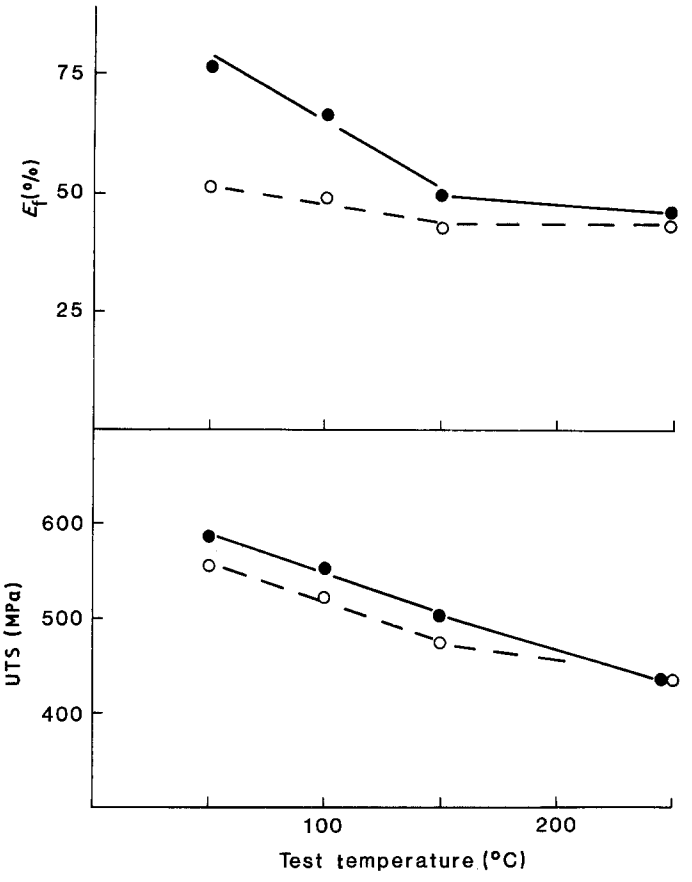


Figure 4 The effects of the test temperature on mechanical properties of a Type 304 steel stressed to destruction at a strain rate of 5.5×10^{-2} sec^{-1}, in (●) rubidium, and (○) argon.

summarized in Table II. There was a slight decrease, from 51 to 43%, in the E_f values for tests in argon and the associated UTS values decreased from 547 to 436 MPa. The effects of the test temperature on mechanical properties in rubidium were greater; the E_f values decreased from 77 to 47% and the UTS values from 674 to 436 MPa. Most of these decreases occurred at test temperatures up to 150° C.

The fractography of the samples was examined using scanning electron microscopy. As might be expected, all the fracture surfaces were heavily dimpled, as illustrated in Fig. 5. There was no obvious effect of test temperature or environment on the nature or scale of the fracture features. Optical microscopy of some cross-sectional samples suggested that more martensite had been formed by samples tested in rubidium or at low temperatures, but no quantification was possible.

These data advance our understanding of the effects of rubidium environments very little. The values for a

TABLE II Mechanical property data

Test temperature (° C)*	Strain rate (sec^{-1})	Environment	UTS (MPa)	E_f (%)
50	5.5×10^{-1}	Ar	567	50.7
50	5.5×10^{-2}	Ar	557	51.3
50	1.1×10^{-3}	Ar	575	65
50	5.5×10^{-5}	Ar	562	72.6
50	5.5×10^{-1}	Rb	596	60
50	5.5×10^{-2}	Rb	584	76.6
50	1.1×10^{-3}	Rb	576	71.7
50	5.5×10^{-5}	Rb	558	78
100	5.5×10^{-2}	Ar	522	49
100	5.5×10^{-2}	Rb	551	66.7
150	5.5×10^{-2}	Ar	477	43.3
150	1.1×10^{-3}	Ar	478	47.3
150	5.5×10^{-5}	Ar	478	44.3
150	5.5×10^{-2}	Rb	501	50.0
150	1.1×10^{-3}	Rb	480	47.3
150	5.5×10^{-5}	Rb	488	50.0
250	5.5×10^{-2}	Ar	436	42.6
250	5.5×10^{-2}	Rb	436	46.7

* ± 2° C.

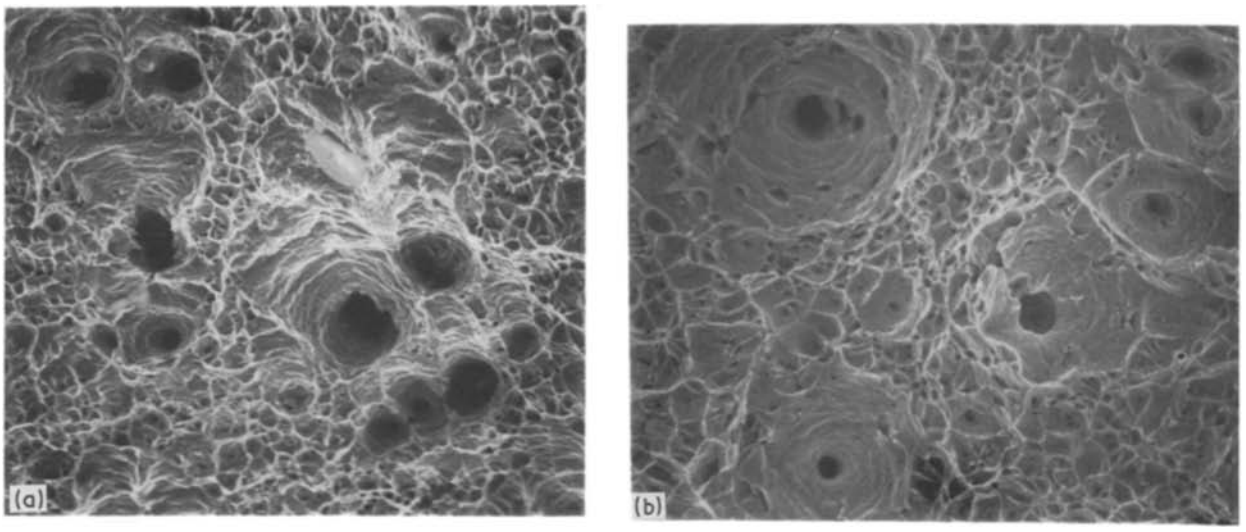


Figure 5 Scanning electron micrographs of the fracture surfaces of samples stressed to destruction at a temperature of 50° C and a strain rate of 5.5 × 10^{-2} sec^{-1} in (a) rubidium and (b) argon, × 760.

test temperature of 50° C confirm the earlier observations, and the diminishing beneficial effect of rubidium as the test temperature is raised is consistent with the earlier rejection of control by a chemical interaction.

3.2. Strain rate effects

At a temperature of 50° C, decreasing the strain rate one thousand-fold from 5.5 × 10^{-2} to 5.5 × 10^{-5} sec^{-1} had only a slight effect on the mechanical properties of samples tested in rubidium, as illustrated by the stress–elongation graphs in Fig. 6 and the data presented in Table II. The effect of strain-rate decrease was much more marked for the argon ductility data – the E_f values increasing from 51.3 to 72.6% – but there was no significant effect on the UTS values.

The effect of increasing the test temperature to 150° C was to diminish any strain rate effects (Fig. 7). Once more the effects of a thousand-fold decrease in the strain rate on the mechanical property data generated by tests conducted in rubidium were negligible. The argon ductility data increased slightly but the UTS values were unchanged by the rate decrease.

On completion of this series, two additional tests were conducted at a test temperature of 50° C using the very fast strain rate of 0.55 sec^{-1}. The behaviour of the sample strained in argon differed little from that previously reported for the test conducted at 0.055 sec^{-1}. However, the ductility of the sample strained in rubidium was significantly lower than that observed earlier during tests using slower strain rates.

Once more, scanning electron and optical microscopy of fracture surfaces and cross-sections failed to reveal any quantifiable effects of environment or changes in the strain rate or test temperature.

Thus with the exception of the 0.55 sec^{-1} test, the mechanical property data generated using rubidium environments were insensitive to strain rate effects. In contrast, the ductility of samples tested in argon was decreased at the low test temperature, 50° C, when the

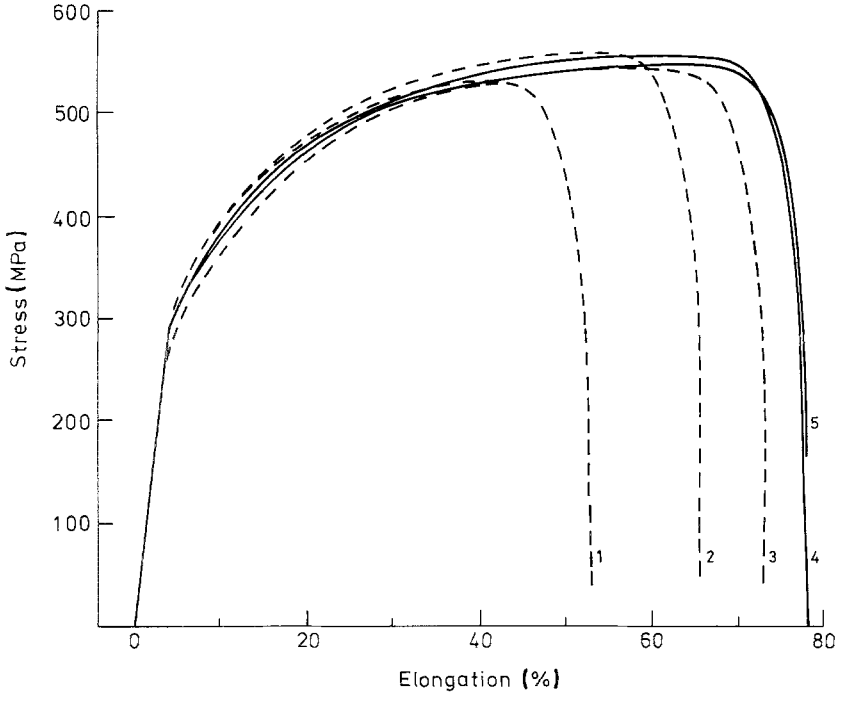
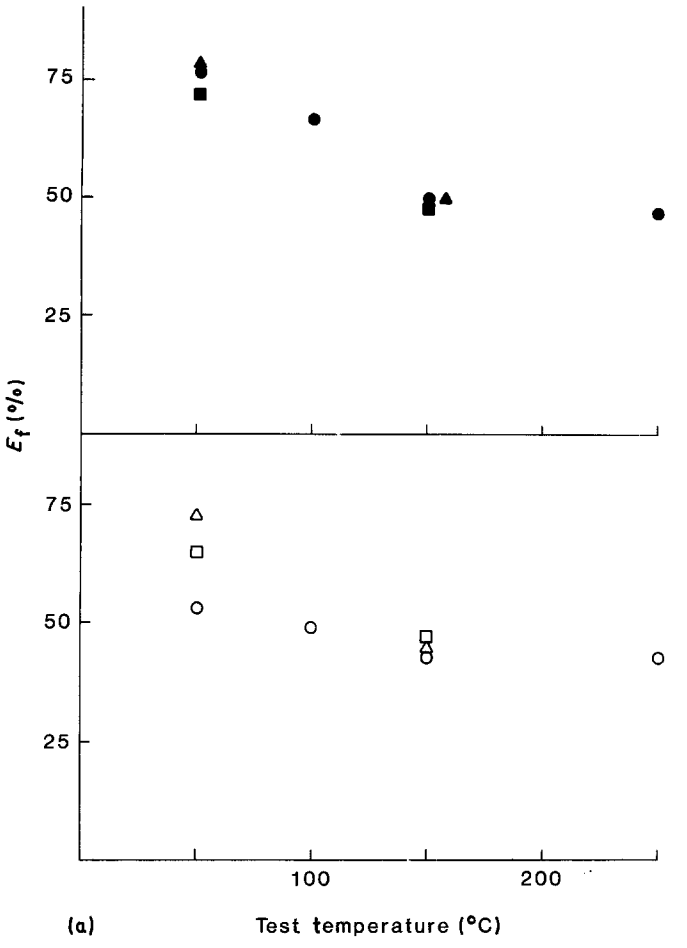


Figure 6 The effect of strain rate changes on the stress–elongation graphs of samples tested at 50° C. (The stresses were calculated by dividing the applied load by the original cross-sectional areas of the samples.) Environments and strain rates (sec^{-1}): (1) Ar, 5.5 × 10^{-2}; (2) Ar, 1.1 × 10^{-3}; (3) Ar, 5.5 × 10^{-5}; (4) Rb, 5.5 × 10^{-2}; (5) Rb, 5.5 × 10^{-5}.



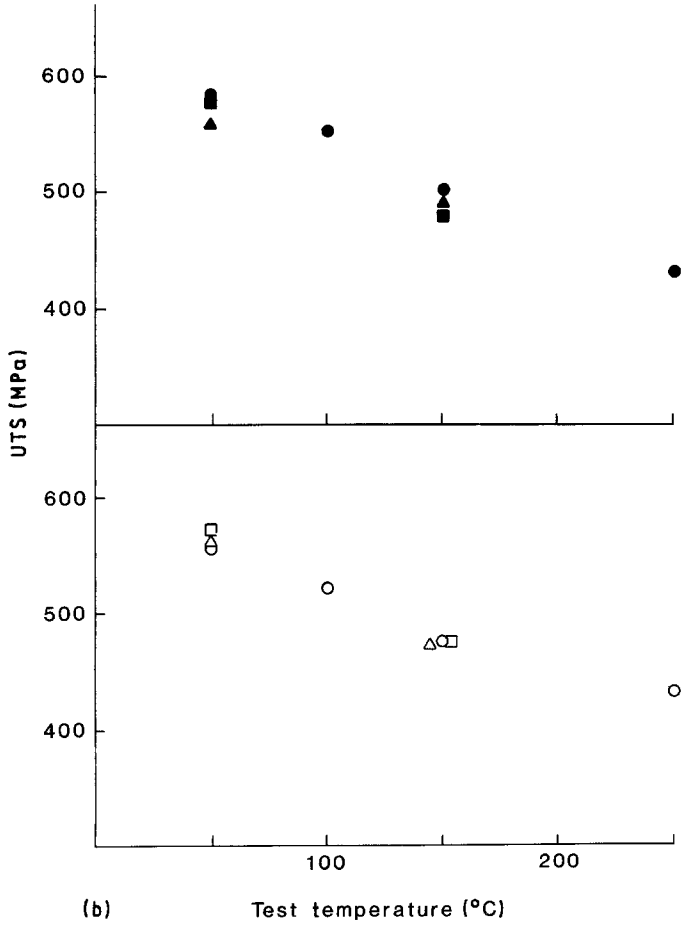


Figure 7 The effect of test temperature and strain rate on (a) E_f and (b) UTS values for a Type 304 steel. Filled symbols, rubidium; open symbols, argon. Strain rates (sec^{-1}): (●, ○) 5.5×10^{-2}; (■, □) 1.1×10^{-3}; (▲, △) 5.5×10^{-5}.

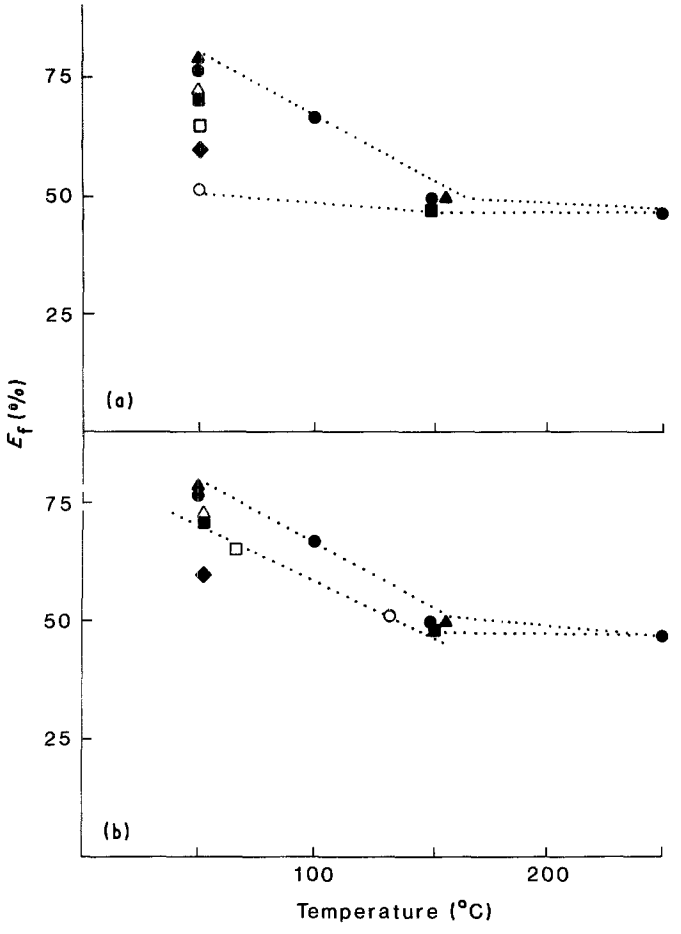


Figure 8 The effect of (a) test temperature and (b) sample temperature on the ductility of a Type 304 steel. Note that the plots exclude argon environment data for test conditions for which sample temperature measurements are not available. Filled symbols, rubidium; open symbols, argon. Strain rates (sec^{-1}): (◆) 5.5×10^{-1}; (●, ○) 5.5×10^{-2}; (■, □) 1.1×10^{-3}; (▲, △) 5.5×10^{-5}

strain rate was increased, and also decreased during low strain rate ($5.5 \times 10^{-5}\,sec^{-1}$) tests when the test temperature was increased. These similar results of increasing temperature and strain rate suggest that fast straining caused the samples to become hot.

It is clear that the ductility of the steel generally decreases as the temperature is raised form 50 to 150° C, and therefore any additional heating of steel being tested in that temperature range is detrimental. A higher temperature caused by fast straining would result in less extensive formation of deformation-induced martensite [7] and hence to a lower work-hardening rate and earlier failure by necking. Any such heating effect should be less marked in rubidium because the thermal conductivity of the liquid metal is about a thousand times that of argon [8]. The result of this greater conductivity could account for the similar behaviour of samples tested in argon at the slowest strain rate of $5.5 \times 10^{-5}\,sec^{-1}$ and samples tested in rubidium at strain rates of up to $5.5 \times 10^{-2}\,sec^{-1}$.

It is difficult to predict how great might be the test-piece temperature rises in the two environments. However, a crude estimate of the maximum possible rise can be made by assuming that all the work of plastic deformation is used to heat adiabatically the gauge section of the sample [9]. This yields an estimate of more than 90° C, a very large and significant value in view of the mechanical effects caused at slow strain rates when the test temperature is raised 100° C from an initial 50° C (Figs 4 and 7).

3.3. Sample temperatures

Efforts were made to measure sample temperature rises during tensile testing because of their possibly major effect on mechanical property data. Two types of experiment were conducted. In the first, a thermocouple was tied to the gauge section of a sample, and in the second a 0.6 mm diameter hole was bored longitudinally into the gauge section of a sample and a 0.5 mm diameter thermocouple was inserted.

When tests were conducted in rubidium using a test temperature of 50° C and a strain rate of $5.5 \times 10^{-2}\,sec^{-1}$, the temperature recorded by the tied-on thermocouples rose by much less than 5° C. In argon environments, their temperatures rose by 10 to 15° C. The temperature rises recorded by the internal thermocouples – which were in better thermal contact with the samples – was only 1° C during rubidium tests, but was up to 82° C when argon was used (Table III). The temperature rises in argon were smaller at slower strain rates: 13 and 2° C at 1.1×10^{-3} and $5.5 \times 10^{-5}\,sec^{-1}$, respectively.

While not definitive, these data support the earlier speculations based on the strain-rate effects. The experimental measurements demonstrate that large temperature rises can occur in samples tested in argon using fast strain rates. The temperature rises are so large that the use of the test temperature to correlate with mechanical property data obtained in argon atmospheres using fast strain rates is incorrect. The sample temperatures should be used. For tests conducted at slow ($5.5 \times 10^{-5}\,sec^{-1}$) strain rates in argon and for strain rates of up to $5.5 \times 10^{-2}\,sec^{-1}$ in rubidium, it is probably adequate to equate the test temperature and the sample temperature. Original and revised plots of the ductility data as a function of

test and measured and assumed sample temperatures are shown in Fig. 8. The effect of taking account of sample temperatures is to bring the data for the two environments closer.

The E_f values for samples strained in argon at 5.5×10^{-2} and $1.1 \times 10^{-3}\,sec^{-1}$ at a test temperature of 50° C no longer fall well below the corresponding data for rubidium environments. They lie within a scatter band of ±3% defined by 5.5×10^{-2}, 1.1×10^{-3} and $5.5 \times 10^{-5}\,sec^{-1}$ strain rate data for rubidium environments at test temperatures of 50, 100 and 150° C. Thus the apparent beneficial effect of rubidium environments illustrated in Fig. 1 is an artefact for the BS 304 S15 steel and may be also for the others. It now seems that rubidium provides an innocuous environment, but it may have an effect on mechanical behaviour if this is limited by the heat transfer characteristics of the test environment.

The one anomalous result remaining in Fig. 8 is that for a sample tested in rubidium at a strain rate of $0.55\,sec^{-1}$. That sample was being strained for only 0.9 sec, and it may be that heat could not be removed by the rubidium fast enough to maintain the sample temperature at 50° C. Fig. 8 suggests that the temperature of that sample could have risen to over 100° C, but there is no supporting evidence.

4. Conclusions

1. Further studies with 304 steel tensile samples have confirmed the rubidium-induced ductility enhancement observed previously at a test temperature of 50° C and a strain rate of $5.5 \times 10^{-2}\,sec^{-1}$. The enchancement decreases as the test temperature is raised and is insignificant at test temperatures of 150° C and above.

2. Decreasing the strain rate to $5.5 \times 10^{-5}\,sec^{-1}$ when using a test temperature of 50° C has no significant effect on the ductility of samples tested in rubidium. The ductility of samples similarly tested in argon increased, and when a strain rate of $5.5 \times 10^{-5}\,sec^{-1}$ was used it approached that obtained in rubidium. Similar effects are observed when a test temperature of 150° C is used.

3. Increasing the strain rate to $0.55\,sec^{-1}$ had no effect on the ductility of a sample in argon at a test temperature of 50° C. The ductility of a sample similarly tested in rubidium decreased.

4. Temperature rises of up to 82° C can occur with samples strained at $5.5 \times 10^{-2}\,sec^{-1}$ in argon at 50° C. The temperature rise is only 13° C if a strain rate of $1.1 \times 10^{-3}\,sec^{-1}$ is used, and is less than 2° C of a rate of $5.5 \times 10^{-5}\,sec^{-1}$ is used. Temperature rises of less than 2° C were observed for samples strained in rubidium at 5.5×10^{-2}, 1.1×10^{-3} and $5.5 \times 10^{-5}\,sec^{-1}$ at a test temperature of 50° C.

5. Ductility differences between samples tested in argon and rubidium largely disappear when the comparison takes account of sample temperatures. The additional data generated in this work suggest that rubidium has a negligible rather than beneficial effect on the mechanical properties of BS 304 S15 steel.

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