

ELEVATED TEMPERATURE TENSILE DUCTILITY MINIMA IN  
TYPES 304 AND 316 STAINLESS STEEL

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## INTRODUCTION

Rhines and Wray [1] pointed out that a minimum in ductility occurs at an intermediate temperature range in all ductile metals and alloys. Several examples cited [1] were copper, cartridge brass, muntz metal, nickel, Inconel, Monel, Hastelloy, Armco iron, 18-8 stainless steel, titanium, aluminum, molybdenum, tantalum, vanadium, lead, and tin alloys.

A typical characteristic of this minimum is a rapid drop in total elongation and reduction of area at a test temperature in the neighbourhood of  $0.5-0.6 T_m$ . Values of these ductility minima frequently fall below 10%, as measured by elongation. Rhines and Wray [1] postulated that at low temperatures, fracture occurs by the usual transgranular crack propagation mechanism, and ductility is high. At temperatures near the minimum, deformation occurs by grain-boundary sliding, and the intergranular cavities formed at triple junctions grow rapidly, causing drastic loss in ductility. At high temperatures, recrystallization occurs simultaneously with intergranular cavity formation continuously breaking up the intergranular fracture path. The ductility increases again.

The first report on the ductility minimum in 18-8 stainless steel was by Newell [2] in 1933. Newell investigated the elevated-temperature tensile reduction of area for stainless steels after four different heat treatments to produce different grain sizes. He found that, although the ductility minimum in 18-8 stainless steel occurs over essentially the same temperature range (850-900°C) for all treatments, the initiation temperature for a rapid drop in ductility decreases with increasing grain size. Furthermore, coarse grain material showed a significantly lower ductility than hot-rolled fine grain material. For example, ductilities at 600°C for fine and coarse grain material were 60 and 38% whereas at 700°C they were 62 and 20%, respectively.

Austenitic stainless steels are used extensively in the construction of liquid-metal fast breeder reactors and will be subjected to elevated temperatures for long periods of time. Therefore, it is important to determine the factors and interrelationships concerning the occurrence of this minimum, including the effects of long-term thermal exposure. It is the purpose of this paper to:

- 1) illustrate conditions under which a ductility minimum can occur;
- 2) illustrate effects of long-term thermal aging on ductility;
- 3) explain results of thermal aging in terms of precipitation and microstructural changes.

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## EXPERIMENTAL DETAILS

Experimental data presented in this paper are from 25 and 51mm thick plates from a single heat of type 304 [3,4] and a 16mm thick plate of type 316. Chemical analysis and grain sizes are listed as follows.

| Type             | Chemical Element, wt % |       |       |       |      |       |      |      |       |       |
|------------------|------------------------|-------|-------|-------|------|-------|------|------|-------|-------|
|                  | C                      | N     | P     | Ni    | Mn   | Cr    | Si   | Mo   | S     | Sl    |
| 304 <sup>a</sup> | 0.047                  | 0.031 | 0.029 | 9.58  | 1.22 | 18.50 | 0.47 | 0.10 | 0.012 |       |
| 316 <sup>b</sup> | 0.065                  | 0.031 | 0.022 | 13.86 | 1.88 | 16.46 | 0.51 | 2.44 | 0.018 | <0.02 |

<sup>a</sup>Laboratory annealed for 0.5 hr at 1093°C. Grain sizes for 25 and 51mm plate were 230 and 280 $\mu$ m, respectively.

<sup>b</sup>Laboratory annealed for 0.5 hr at 1065°C. Grain sizes for 16mm plate for the as-received and reannealed conditions were 50 and 90 $\mu$ m, respectively.

The test specimens were threaded-end bars having a gage diameter of 6.35mm and reduced section of 31.8mm. Specimens were lathe machined, cleaned, and subjected to the following heat treating conditions:

- 1) As-received or mill-annealed - contains residual cold work [5] due to thermomechanical processing.
- 2) Reannealed or laboratory annealed - consists of holding at 1093°C for types 304 and 1065°C for type 316 for 0.5 hr followed by fast cooling.
- 3) Thermally aged - consists of thermal exposure of specimens at the test temperature for varying times (7 to 10,000 hr).

Tensile tests were performed in air at temperatures in the range from room temperature to 760°C and strain rates from  $1 \times 10^{-4}$  to  $5 \text{ min}^{-1}$ . One test at 482°C was performed at  $2.6 \times 10^{-6} \text{ min}^{-1}$ . The strain rate was controlled by crosshead speed. Additional details of testing methods can be found elsewhere [3,4]. Experimental results presented in this paper also include data reported by Steichen [6,7].

## RESULTS

Three-dimensional computer constructed plots of total elongation and reduction of area for 25 and 51mm plates of type 304 stainless steel are presented in Figure 1. Shallow perturbations in ductility surfaces resulted from combining the data from 25 and 51mm plates. It may be noted that both measures of ductility exhibit minima. However, the minimum value trends are not as pronounced for total elongation. The influence of strain rate on the initiation temperature (indicated by arrows) for the drop in ductility is more clearly shown in the two-dimensional plots of data in Figure 2(a). A change in strain rate from  $5$  to  $1 \times 10^{-4} \text{ min}^{-1}$  resulted in a shift in ductility drop initiation temperature from 727 to 438°C ( $0.60$  to  $0.42 T_m$ ), where  $T_m$  is the melting point  $1673 \text{ K}$ . Ductility at the minimum decreased with decreasing strain rate and reduction of area reached a value of 22% at  $\dot{\epsilon} = 1 \times 10^{-4}$  for type 304 stainless steel. Figure 2(b) shows that for two heats of type 304 the ductility loss initiation temperature was lower for the coarse grain heat as observed in other materials [8]. This may also be partially associated with slight differences in compositions of the two heats [9].

Similar results were observed for type 316 and, although data were not sufficient to develop the three dimensional plots for a single material, a minimum was observed, Figure 2(c). As shown in Figure 2(c), the minimum in the reduction of area appears to be influenced by grain size and possible composition differences.

Thermal aging for periods exceeding 1000 hr at 649°C can eliminate the ductility minimum for types 304 and 316 stainless steel at 649°C and strain rate of about  $2 \times 10^{-3} \text{ min}^{-1}$  (see Figures 2(b) and (c)). However, thermal aging at lower temperatures results in some decrease in ductility, Figures 2(b) and (c) [7,10] in comparison with reannealed material. The minimum value of 649°C for aged type 304 is 55%, even after an aging period of 10,000 hr, as opposed to 22% for the unaged material.

Optical microstructural analysis was performed [11,13] on specimens of type 304 tested at 649°C and three different strain rates, 0.05, 0.005, and  $0.0005 \text{ min}^{-1}$ . All specimens showed grain boundary cracks. Estimates of the number and mean orientation (major axis) of these cracks on the same longitudinal specimen plane area with respect to the stress axis are indicated in Figure 2(a). The density of cracks increased towards the fracture zone for the three strain rates. The specimen tested at  $0.005 \text{ min}^{-1}$  exhibited the largest fraction of cracks longer than one grain facet. The crack orientation with respect to the stress axis changed from  $50^\circ$  to  $80^\circ$  at the two lower strain rates. Specimens tested at 649°C and at a strain rate of  $0.005 \text{ min}^{-1}$  also showed some cracking at annealing twin boundaries.

Optical microstructural analysis was also performed on type 316 stainless steel specimens tested at a single strain rate ( $0.005 \text{ min}^{-1}$ ). Three different test temperatures were used: below the ductility minimum, at the minimum, and beyond. The following are the observations on these specimens:

- 1) Below minimum (593°C) - Necking was substantial. Grain boundary crack density was low. Fracture was transgranular and occurred along the maximum shear stress plane in the neck. There was extensive intragranular deformation.
- 2) At minimum (649°C) - The necking was limited and grain-boundary cavitation was extensive. Fracture was predominantly intergranular with only limited intragranular deformation.
- 3) Beyond minimum (704°C) - Necking was extensive. Fracture appeared transgranular with extensive inter- and intragranular deformation.

Metallographic findings on unaged specimens appeared consistent with the model proposed by Rhines and Wray [1] for the ductility minimum in metals and alloys. That is, the ductility minimum was associated with temperature, strain rate, and metallurgical conditions under which cracks propagation was uninhibited.

Austenitic stainless steels can undergo precipitation reactions during elevated temperature testing and exposure [14-18]. Indeed this may be the reason why the reannealed type 316 stainless steel regains its ductility at temperatures above about 650°C (Figure 2(c)). These precipitates form at grain boundaries, twin boundaries (incoherent and coherent) and on dislocations and can influence the mechanical properties [10]. For example, Driver [19] has shown that  $M_{23}C_6$  precipitates formed by aging at 700°C can retard grain boundary sliding and migration and thereby introduce a transgranular failure mode during fatigue testing. Sikka et al. [20] have shown that thermal aging at 649°C can prevent grain boundary

cracking in type 304 and promote extensive intragranular deformation and high ductility at 593 and 649°C. The transgranular failures appeared in some instances to be associated with annealing twin boundary cracks. The observation of annealing twin boundary cracks and the absence of grain boundary cracks was suggested to result from a combination of several factors influenced by precipitation; for example, relative intra- and intergranular strength, high cohesion strength of large particles within austenitic grain boundaries, high strength of particles, and the possibility of grain boundary migration between the large particles. The decrease in ductility as a result of carbides formed at lower temperatures is considered as the consequence of small particle size and spacing between the particles. Thus, it appears that thermal aging modifies the characteristics of intergranular crack formation and failure. Residual and major element content differences may also be a major factor as well in that differences in ductility behaviour are apparent when comparisons are made between types 304 and 316 (Figure 2(a) and (c)) at a strain rate of about 0.005 min<sup>-1</sup>.

#### CONCLUSIONS

Present investigation has confirmed the existence of a ductility minimum in austenitic stainless steels of types 304 and 316. Strain rate, grain size, and slight composition differences influenced the ductility loss initiation temperature. Thermal aging at 649°C eliminated the ductility minimum. Aging at lower temperatures still results in some lowering in ductility in comparison with reannealed material. The effects of thermal aging were associated with carbides modifying the characteristics of intergranular crack formation and growth. Although austenitic stainless steels are known for their high ductility and toughness, they do have a range of diminished ductility at elevated temperatures particularly at coarse grain sizes and this should be considered in their application.

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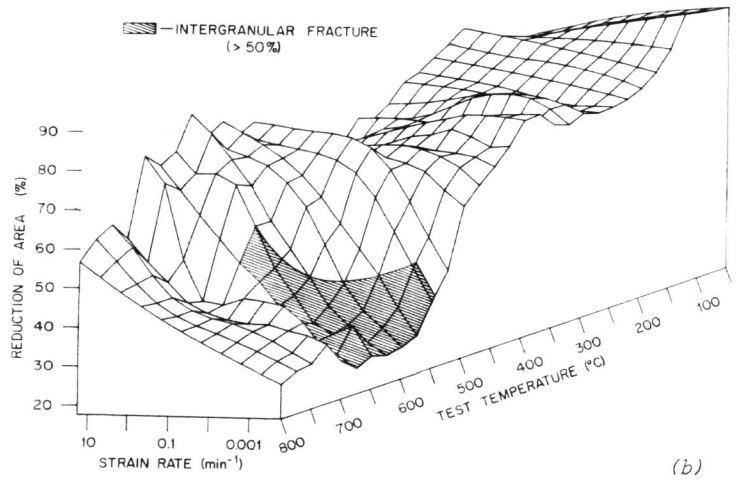
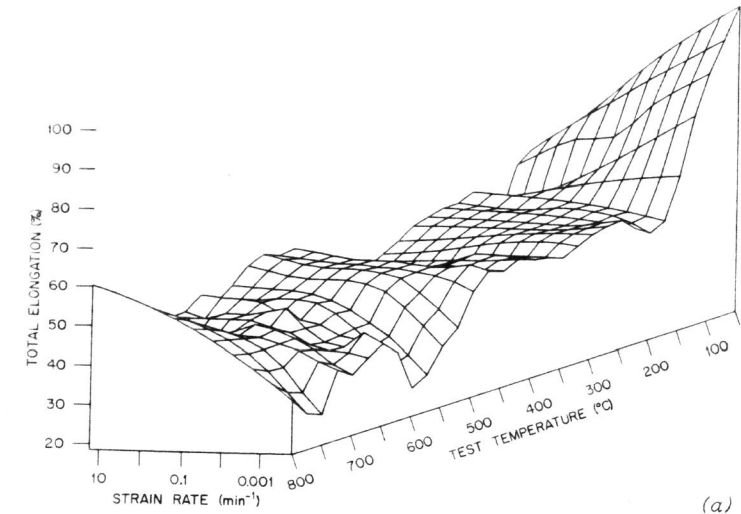


Figure 1 Three Dimensional Plots of Total Elongation and Reduction of Area as a Function of Log Strain Rate and Test Temperature for 25 and 51 mm Plates of Type 304 Stainless Steel. The Hatched Area in Figure 1b refers to Intergranular Fracture > 50%

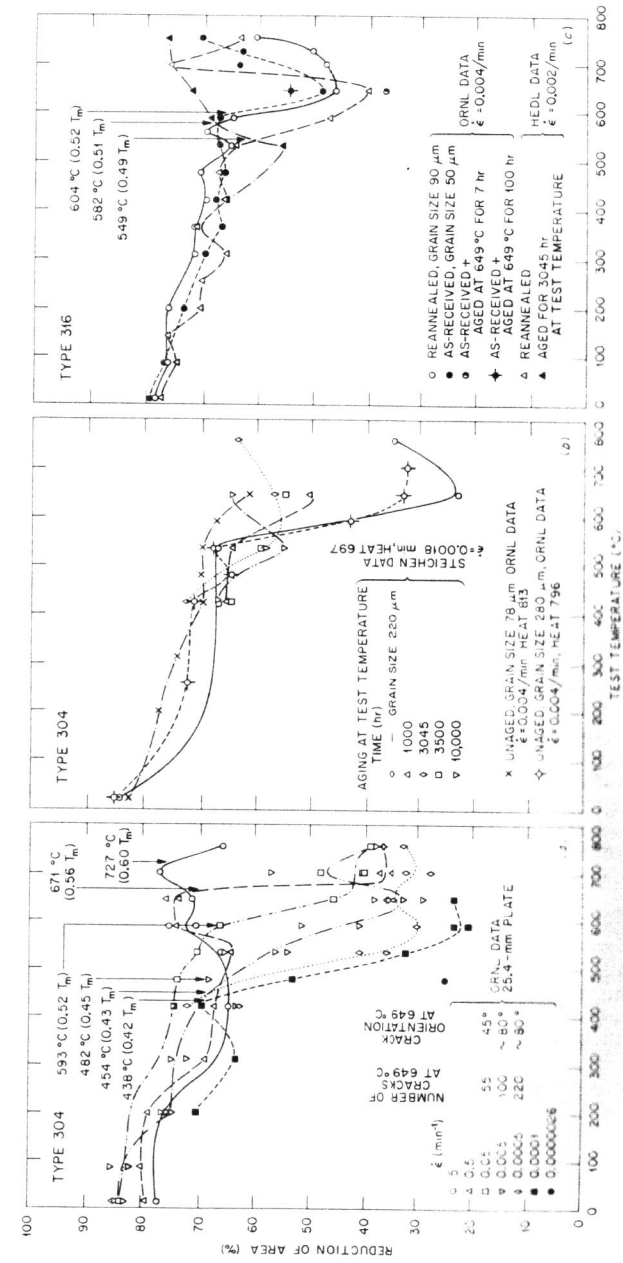


Figure 2 Tensile Reduction of Area as a Function of Test Temperature for Types 304 and 316 Stainless Steel.  
 (a) Effect of Strain Rate for a Single Heat of Type 304  
 (b) Effect of Grain Size and Aging on Type 304  
 (c) Effect of Grain Size and Aging on Type 316