## Superplastic Behavior of AISI 329 Duplex Stainless Steel at High Temperatures under Tensile Loading

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## Abstract

This study examined the hot tensile behavior of AISI 329 duplex stainless steel to specify its superplastic conditions. Tensile tests were carried out at temperatures between 900 and 1200 °C under three strain rates of 0.15 S<sup>-1</sup>, 0.04 S<sup>-1</sup>, and 0.001 S<sup>-1</sup>. The stress-strain curves are associated with yield point phenomenon under two strain rates of 0.15 S<sup>-1</sup> and 0.04 S<sup>-1</sup> at temperatures between 950 and 1200 °C. Strain softening is a predominant feature at temperatures above 1000 °C. No evidence of recrystallization was observed by metallugraphic studies, also multistage tensile tests showed no strong evidence of recrystallization (i.e., the rate of strain softening was very low). Microstructural studies showed that the amount of ferrite phase increases by increasing the test temperatures above 1000 °C associated with elongations of up to 400 percent. The maximum elongation occurs at 1050 °C and elongation decreases by subsequent increase in temperature. This seems to be due to the increment of ferrite phase at high temperatures, since increasing ferrite phase results in a sharp decline in workability of these steels.

**Keywords:** Superplastic Behavior, Duplex Stainless Steel, Grain Boundary Sliding, Recrystallization, High Temperatures, Tensile Loading.

### 1. Introduction

Duplex stainless steels (DSS) with excellent corrosion resistance and yield stress are favorable materials for many applications. Nevertheless its main limitation is low hot workability due to the generation of edge cracks [1]. Considerable studies have been conducted to investigate hot workability of DSS, where deformation mechanisms depends on the individual phase, temperature and strain rate [2]; it has been reported that while austenite undergoes dynamic recrystallization during hot deformation, dynamic recovery occurs in ferrite [3], although there are some

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contradicting reports where no sign of dynamic recrystallization has been observed [4, 5, 6, 7]. Some researchers have noticed the tendency of superplastic behavior in DSS [2, 3, 8, 9, 10, 11]. Grain boundary sliding has been reported to be the dominant mechanism involved [12], thus indicating the importance of fine grain structure together with suitable strain rate sensitivity coefficient (m).

Generally, in most of the studies conducted so far, no specific analysis has been reported on the precise mechanism of superplastic deformation at high temperatures, stress-strain behavior of these steels, and the metallographic differences at various temperatures and strain speeds, and the studies have just estimated the value of 'm' coefficient. For instance, Lee et al., found that steel type 2205 DSS strip exhibited superplastic-like deformation behavior under continuous annealing condition (at 1025 to 1100 °C and  $2.4 \times 10^{-5}$  to  $5.4 \times 10^{-5}$  S<sup>-1</sup>) with a high m-value that is 0.533 [13]. In another research, a ( $\alpha$ + $\gamma$ ) duplex Fe-25Cr-7Ni-3Mo alloy was examined and it was found that an elongation of more than 800% was obtained at a strain rate of  $5 \times 10^{-2}$  S<sup>-1</sup> at 1323 °K and m-value was found to be 0.5 at the same strain rate [14].

In this work tensile behavior of a DSS at high temperatures and different strain rates have been studied to specify the conditions for superplastic behavior to occur.

### 2. Materials and experiments

The material used in this study is a AISI 329 DSS with the chemical composition given in table 1. The rods were annealed for 1 hour at 1250°C, then slowly cooled. Tensile tests were carried out on 5 mm diameter and 40 mm gage length tensile specimens using a 250 KN computer controlled machine at temperatures between 900 and 1200°C with 50°C intervals. Strain rates were 0.001, 0.04 and 0.15 S<sup>-1</sup> and were kept constant throughout the tests. The etchant used for metallugraphic examinations was a solution of 100 ml Hcl plus 100 ml etanol and 5 gr copper chloride in 100 ml distilled water. A computer equipped with an image analyzer and a quantometer was used for phase analysis and determining the chemical composition. To measure the average size of grains, one hundred grains were randomly selected by computer controlled image analyzer and their diameter in directions parallel and perpendicular to the rolling direction were measured and the average values where determined. Subsequently, the mean values of the diameter with the corresponding standard deviation were obtained.

Table 1) Chemical Composition of AISI 329 DSS Steel.

| С    | Fe      | Cr | Ni | Mn   | Si   | Mo  | V    | W    | Al   | Cu  | Ti   | Р     | S     |
|------|---------|----|----|------|------|-----|------|------|------|-----|------|-------|-------|
| 0.12 | Balance | 21 | 5  | 0.32 | 0.55 | 0.2 | 0.04 | 0.03 | 0.16 | 0.1 | 0.73 | 0.024 | 0.007 |

#### 3. Results and Discussion

A typical microstructure of the annealed rods is shown in Fig. 1.



Figure 1) Microstructure of the studied DSS after Anneal.

The microstructure consists of austenite (white phase) and ferrite (dark phase). The mean value of grain size and the standard deviation value  $are19 \times 8 \mu m^2$ , 18.36 respectively. Phase analysis indicates that the microstructure consists of 70% ferrite and 30% austenite. Stress-strain curves of the DSS used in this study are shown in Figs. 2 to 4. By increasing temperature, under a constant strain rate, flow curves shift to lower stresses. Strain softening is observed in all temperatures except those tested at 900°C under strain rates of 0.04 and 0.15 S<sup>-1</sup> and at 900°C under the strain rate of 0.15 S<sup>-1</sup>. At higher temperatures the slope of the curves decreases and most parts of the curves become linear; it seems that the dynamic recovery is the dominant mechanism involved, as has been reported in previous studies [8, 15, 16]. In the stress-strain curves obtained at temperatures above 900°C under strain rates of 0.04 S<sup>-1</sup> and 0.15 S<sup>-1</sup> yield point phenomenon is clearly visible.

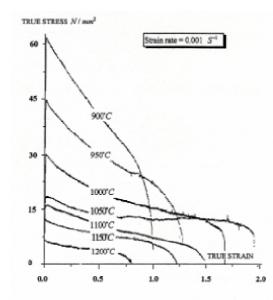


Figure 2) True strain-stress curves at different speeds and in 0.001s<sup>-1</sup> strain speed.

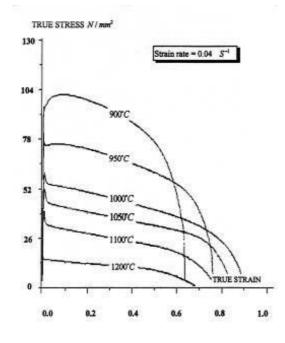


Figure 3) True strain-stress curves at different temperatures and at 0.04 s<sup>-1</sup> strain speed.

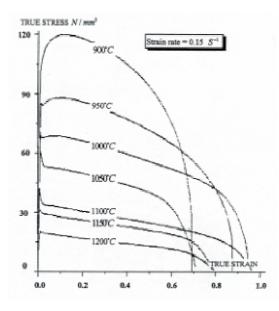


Figure 4) True strain-stress at different temperatures and at 0.15 s<sup>-1</sup> strain speed.

The amount of yield point drop is small at low temperatures and then increases to a maximum value by increasing temperatures up to  $1100^{\circ}$ C and declines by a further increase in temperature to  $1200^{\circ}$ C. The result of multi state tensile test at  $1100^{\circ}$ C conducted with unloading intervals of 25, 60 and 120 seconds (Fig. 5) also reveals a strong yield point behavior in this steel.

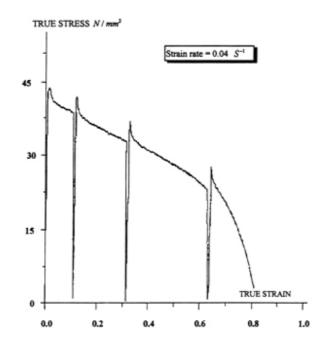


Figure 5) Multi phases strain-stress curves at1100°C and 0.04 s<sup>-1</sup> strain speed.

The interesting point is that by increasing unloading time to 60 seconds and even further to 120 seconds at 1100  $^{\circ}$ C, no change in the amount of yield drop was observed. The exact reason for this phenomenon is unknown for the authors; perhaps the interaction of titanium atoms with dislocations could cause this effect [17]. Although at this high temperatures the mobility of titanium atoms are high, it would be surprising for substitutional titanium atoms to be able to generate a contracted atmosphere to cause such a yield drop, like those observed in the case of interstitial atoms. Further studies are required to verify this point.

As it is seen in Figs. 2, 3, 4 elongation of the specimens tested at temperatures around  $1050^{\circ}$ C reach to a maximum value of %380. This effect is more pronounced for the lowest strain rate (i.e. 0.001 S<sup>-1</sup>). It seems the increase in elongation is associated with superplastic behavior, involving grain boundary sliding in addition to dynamic recovery. It is expected that by increasing the temperature, the tendency for grain boundary sliding will also increase, thus increasing the ductility (i.e. elongation). However, as it is illustrated in Fig. 6, the variation of elongation with temperature after reaching to a maximum value which is dependent on the strain rate, declines by a further increase in temperature.

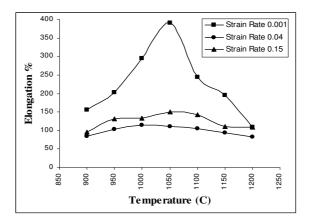


Figure 6) Elongation percent curves according to the temperature for three different strain speeds.

Figs. 7, 8, 9 and 10 illustrate the microstructure of the deformed specimens at temperatures of 900, 950, 1000, 1050, 1100, 1150 and 1200°C and quenched in water respectively;

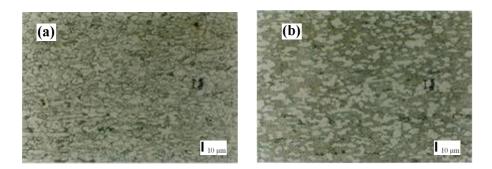


Figure 7) Metallugraphical pictures of 0.001 s<sup>-1</sup> strain speed at different temperatures; a)  $900^{\circ}$ C, b)  $950^{\circ}$ C.

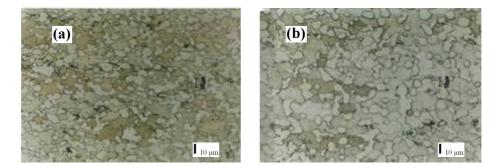


Figure 8) Microstructure of 0.001 s<sup>-1</sup> strain speed at different temperatures; a) 1000 $^{\circ}$ C, b) 1050 $^{\circ}$ C.

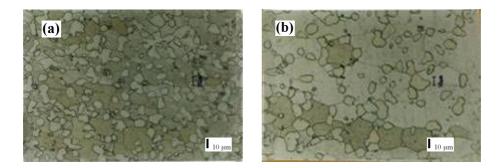


Figure 9) Metallugraphical pictures of 0.001 s<sup>-1</sup> strain speed at different temperatures; a)  $1100^{\circ}$ C, b)  $1150^{\circ}$ C.

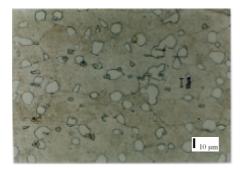


Figure10) Metallugraphical picture of 0.001 s<sup>-1</sup> strain speed at 1200°C.

The amount of ferrite phase increases by increasing the temperature of the deformation. The corresponding result of phase analysis of ferrite and austenite constituent phases is shown in Fig. 11; the rate of the increment in ferrite phase accelerates at higher temperatures. This view is confirmed by studying the pseudo-binary phase diagram seen in Fig. 12. Several studies have shown that the grain boundary sliding is more favorable in austenite-austenite boundaries than austenite-ferrite boundaries and the former is more favorable than ferrite-ferrite grain boundaries [15, 18, 19]. In addition, the ductility of the ferrite phase at high temperatures is less than austenite. However, since the maximum value of elongation is remarkably strain rate sensitive, it seems that the former is not so effective.

From the obtained stress-strain curves given in Figs. 2, 3 and 4, the strain rate sensitivity coefficient of studied DSS at different temperatures were determined as follows;

The flow stress at a given strain rates may be presented as;

$$\sigma = C \left( \epsilon^{\circ} \right)^{m}$$

(1)

Where; m : strain rate sensitivity C : a constant depending on the material From Equ. (1) it is concluded that; m = Ln  $(\sigma_1/\sigma_2)/Ln (\hat{\epsilon_1}/\hat{\epsilon_2})$ 

Where;

 $\sigma_1$ ,  $\sigma_2$ : corresponding stresses on the flow curves (obtained under strain rates of  $\varepsilon_1^{\circ}$  and  $\varepsilon_2^{\circ}$  respectively) at a certain strain.

Table 2 shows the obtained values of strain rate sensitivity (m-value) of the studied DSS at different temperatures by using Equ. (2) in this study. As shown in Table 2, the average strain rate sensitivity value (m) of DSS type AISI 329 is 0.48. It indicate that DSS type AISI 329 steel has a characteristic of superplastic deformation.

 Table 2) Obtained values for strain rate sensitivity coefficient (m) of different temperatures.

| T (°C) | <b>Obtained Values for (m)</b> |       |       |       |       |  |  |  |  |
|--------|--------------------------------|-------|-------|-------|-------|--|--|--|--|
| 1050   | 0.474                          | 0.483 | 0.489 | 0.481 | 0.471 |  |  |  |  |
| 1100   | 0.469                          | 0.464 | 0.476 | 0.510 |       |  |  |  |  |
| 1200   | 0.458                          | 0.489 | 0.457 |       |       |  |  |  |  |

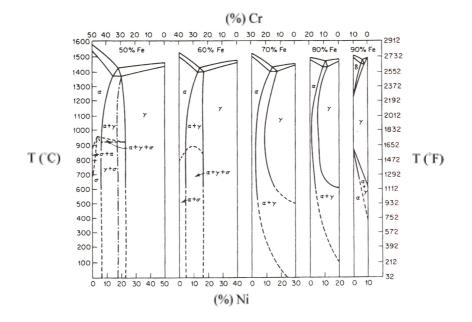


Figure 11) Cross Section of Fe-Cr-Ni System.

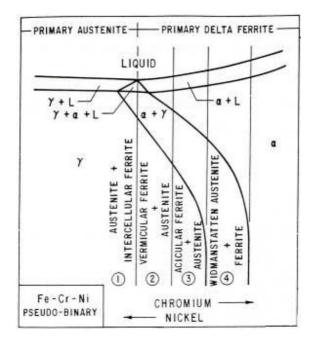


Figure 12) The pseudo-binary phase diagram of Fe-Cr-Ni.

# 4. Conclusion

- Yield point phenomenon was observed at higher strain rates and temperature range of 900-1200°C. The main reason for this phenomenon is the interaction of titanium atoms with dislocations.
- Work softening is predominant at temperatures higher than 1000°C. It seems it was caused by a mechanism based on Austenite-Ferrite boundary sliding that prevents deformation of internal region of Austenite or Ferrite grains.
- Sequential tensile tests and Metallographic tests revealed that dynamic recrystallization was not the principal softening mechanism.
- Metallographic test results showed that Austenite to Ferrite transformation occurred by increasing the test temperature.
- At low strain rates, tensile specimens revealed superplastic behavior, with up to 400 percent elongation.
- By increasing the volume fraction of Ferrite, the ductility decrease markedly.

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