# ARTICLE

# Effect of Widmanstätten Particles on the Flow Behavior of Duplex Stainless Steels at High Temperatures

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#### Abstract

Two duplex stainless steels with different austenite particles distribution inside the ferrite matrix were deformed by torsion at high temperatures. Two different flow curve shapes were observed. The type I indicated that, after rapid loading, the stress rose by an extended linear region, which dropped after overcoming a stress peak. The type II flow curves showed a very fast stress increase during loading, after which the curves displayed extensive work hardening and softening regions. The microstructural evolution was observed, which indicated several differences. However, the most significant finding was the presence of particles of austenite Widmansträten-like structure inside the ferrite in samples that showed the type II flow curves.

Keywords: Duplex Stainless steel, Flow Curves, Widmanstätten Particles.

### Introduction

Duplex stainless steels consisting of austenite particles within a ferrite matrix solidify with a ferritic structure at temperatures of around 1430°C, then changing to a two-phase structure during cooling, with the transformation  $\alpha \rightarrow \gamma$  by solid-state reaction. During hot work processing, these steels are usually reheated to 1250°C and strained on cooling by rolling or forging in multiple pass schedules to temperatures of close to 1000°C. In this temperature range, phase transformation is characterized by the

nucleation and growth of austenite particles inside the ferrite matrix. The presence of a massive second-phase within the matrix heightens the complexity of plastic behavior and the microstructure of the material may limit the amount of attainable strain (1).

In addition to the characteristics of each of the constituent phases, the plastic behavior of duplex stainless steels depends on the nature of the interfaces between phases, the volume fraction, shape and distribution of the austenite particles inside the ferrite matrix, and deformation conditions (temperature and strain rate). During high-temperature straining, after a certain degree of work hardening, ferritic stainless steels soften by intense dynamic recovery, while austenitic stainless steels soften by dynamic recrystallization due to their fairly low stacking fault energies (2,3). Several authors have reported that hot working characteristics are strongly dependent on the volume fraction of austenite (4,5). A decrease in the austenite fraction produces localized strain in the ferrite matrix, which causes increased ductility and reduced strength. Mixing a high volume fraction of massive austenite particles inside the ferrite matrix produces greater gains in a material's strength than straining either phase separately, because the mutual slip constraints harden both phases (6) and the phase morphology retards normal restoration mechanisms (7). Moreover, strain conditions act on plastic behavior since, upon cooling, ferrite produced by solidification becomes transformed into austenite and the variation in temperature affects the volume fraction and the chemical composition of the two phases (8).

Recent studies have shown that, along with deformation mode imposed by mechanical testing, the shape of flow curves is affected by the factors that control the plastic behavior of duplex stainless steels. Iza-Mendia et al. (9.10) observed that flow curves for

wrought material strained by torsion display a characteristic shape: at low strains, there is a parabolic strain hardening that reaches a  $\sigma_o$  value, followed by a stage of almost linear hardening rising to a peak, after which the flow stress decreases continuously to fracture. Balancin et al. (11) found that, when the volume fraction of austenite is small, the presence of austenite particles with a Widmanstätten-like structure produced by a solid-state reaction immediately before straining increases the strength, with the curves showing rapid work hardening to a hump, followed by extensive flow softening.

The purpose of this study was to analyze the effect of austenite particles with Widmansttäten-like structures on the flow curves of duplex stainless steels with a high volume fraction of austenite subjected to hot torsion tests.

#### **Materials and Methods**

Two types of duplex stainless steels, DIN W. Nr. 1.4462 and 1.4463, referred to herein as D1 and D2, were investigated in this work. Their chemical compositions are given in Table I.

Table 1 – Chemical composition of the materials (%W)

Steel	C	Cr	Ni	Mo	Mn	Si	N
D1	0.033	22.2	5.6	3.0	1.8	0.5	0,121
D2	0.054	23.9	7.4	2.3	0.9	1.0	0.136

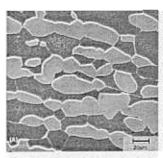
Two different treatments were applied in order to obtain different austenite particle distributions within a ferrite matrix before straining. The first treatment consisted of heating samples of D1 and D2 to 1250°C, holding them at this temperature for 1 hour and then water quenching them. After this initial thermal treatment (TT), some of the D1 samples and all the D2 samples were cold rolled (CR); the D2 samples strained to 30% and D1 strained to 60%. Solid torsion specimens were machined to a 6.5 mm diameter and a 20 mm length in the reduced central gage with the torsion axis aligned in the rolling direction.

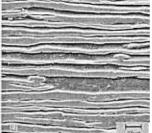
Mechanical tests were carried out on a computerized hot torsion machine described in (12). Specimens were heated to 1000°C and 1100°C at a rate of 180°C/min, maintained at these temperatures for 60 s, and strained to fracture at a strain rate of 1s<sup>-1</sup>. To retain the high-temperature microstructures for further observation, water was injected into a quartz tube surrounding the samples immediately upon

reaching the fracture strain. After standard metallographic procedures, a deposition color etch composed of 100 mL of distilled  $H_2O$ , 20 mL of HCL and 2 g of potassium metabissulfite was applied in order to reveal the microstructure. Microstructural observations were carried out using optical microscopy (OM) and scanning electron microscopy (SEM).

#### Results

Starting microstructures produced by the initial treatments are displayed in Figure 1. These microstructures clearly reveal the changes caused by the different treatments applied. In the D1 samples -(TT), the austenite particles tended to form a dispersed second-phase within the ferrite matrix (Figure 1a). The austenite particles here were not contiguous and did not form layers, as observed in the D1 samples - (TT) and (CR), (Figure 1b). After cold rolling, the microstructure of D1 tended toward a "percolated" type arrangement, in which the second-phase particles were contiguous throughout the microscopic specimen (13). On the other hand, the austenite particles in D2 - (TT) and (CR) showed an intermediary distribution; these particles were not yet contiguous owing to insufficient strain.





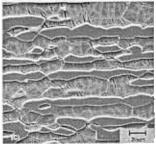


Figure 1 – Starting microstructure: (a) D1 thermally treated (TT); (b) D1 thermally treated (TT) and cold rolled (CR), and (c) D2 thermally treated (TT) and cold rolled (CR).

Figure 2 shows the flow curves obtained from the torsion tests. As can be seen, these curves have two different shapes. In the first set, type I, curves number 2 show that after rapid loading, when plastic strain began, the stress tended to remain constant with a low degree of straining. This was followed by an extensive work-hardening region, which dropped after reaching a peak stress. The curve representing D1 – (TT) and (CR) displays an almost linear rate stage in the work-hardening region at 1000°C while, at 1100°C, the curve indicates a small region with nearly constant stresses after the loading region. It is worth noting, in these curves, that the work-softening region is very small; the ductility of steel D1 is low under these conditions and failure of the samples occurred after little straining.

In the second set of curves, type II (numbers 1 and 3 in Figure 2), the stress shows a very fast increase during loading ( $\varepsilon = 0.05$ ), after which the curves exhibit extensive work-hardening and work-softening regions, with a mild stress peak at an intermediary position. Again, curves number 3 show no work softening region due to the low ductility of this steel under these conditions.

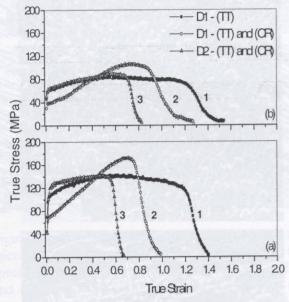


Figure 2 – Flow curves for steels tested at 1000°C (a) and 1100°C (b)

Figure 3 shows microstructures observed after straining by hot torsion. There are several differences between these microstructures. However, the most important difference is the presence of small particles of austenite with Widmansttäten-like structures inside the ferrite matrix in D1- (TT) and D2- (TT) and (CR), (Figures 3a, c, d and f). It should be noted that these samples displayed type II flow curves, while the microstructures of the samples without the presence of Widmansttäten-like particles (Figure 3b and e) showed type I flow curves.

Figure 3 shows that the austenite particles, even those with a Widmansttäten-like shape, tend to align in the direction of strain, suggesting that at least some of the Widmansttäten-like particles were formed during the reheating and hold time prior to the onset of straining. It is also worth noting that, in the samples in which austenite particles were contiguous in the initial microstructure, there is joint rotation and fragmentation of the austenite particles (Figures 3b and e).

A comparison of Figures 1 and 3 shows that the volume fraction of austenite increased during hot torsion tests. Table 2 shows the values measured for initial and final volume fractions of ferrite. Taking into account the margin of experimental error, one can consider that all the samples had the same initial volume fraction of austenite, independently of the initial treatment. The volume fractions of austenite increased after straining. This increase may occur through the formation of new particles with Widmansttäten-like structures or by coarsening of the austenite particles.

Table 2 – Volume fraction of ferrite (%).

Condition	D1 - (TT)	D1- (TT) and (CR)	D2 – (TT) and (CR) $60.2 \pm 2.7$	
Initial	58.3 ± 1.6	55.4 ± 1.4		
1000°C	$26.7 \pm 1.7$	$38.1 \pm 2.9$	$12.4 \pm 1.8$	
1100°C	$36.1 \pm 2.4$	$40.9 \pm 2.5$	$22.1 \pm 2.0$	

#### Discussion

The samples in this study were strained by torsion tests. In this deformation mode, one of the reduced central gage extremities of the sample is rotated around its axis while the other end is fixed. At the beginning of the test, the specimen is subjected to loading, which causes elastic and micro strains. The plastic strain begins in the softer alpha phase and proceeds with the simultaneous straining of both phases. At this stage, the plates of austenite that were aligned along the torsion axis tend to become aligned parallel to the maximum shear stress. The rotation of second-phase particles took place under all the conditions studied here. Details of this process are shown in the micrograph of Figure 4, taken from a D1 sample - (TT) and (CR). It should be noted, from this Figure, that as straining was applied, the austenite particles rotated and the motion of the interfaces gave rise to a gradual fragmentation of the austenite plates. After some straining, the particles were no longer contiguous. As expected, the rotation and fragmentation of austenite particles were more dramatic

on the surface of the sample than in its central axis.

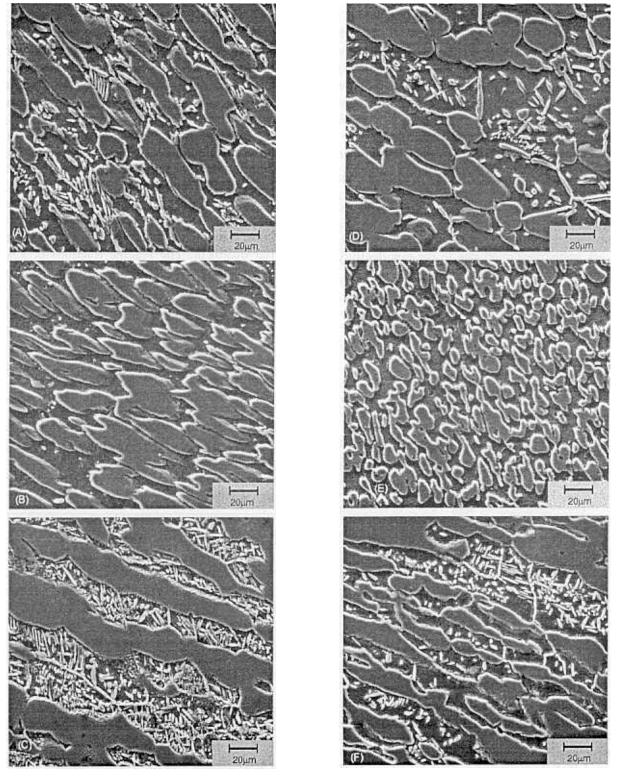


Figure 3 – Microstructures observed by SEM after straining in hot torsion: (a) steel D1 (TT), (b) steel D1 (TT) and (CR) and (C) steel D2 (TT) and (CR) for tests carried out at 1000°C; (d), (e) e (f) at same conditions for tests carried out at 1100°C.

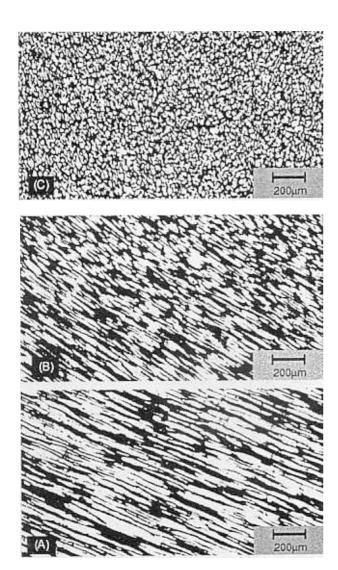


Figure 4 – Micrograph showing the evolution of the microstructure as a function of the radius of the specimen during hot torsion of the sample D1 – (TT) and (CR) at 1000°C. (a) Center of the sample, (b) intermediary position, and (c) surface of the sample.

The flow curves of the D1 samples, (TT) and (CR), exhibit a characteristic shape described in Reference (9). The fast parabolic strain-hardening region at the beginning of the test is associated with elastic and micro strains. The small region of almost constant stress after loading observed at 1100°C can be attributed to the isolated straining of the ferrite matrix at the onset of plastic strain. The region of quasi-linear hardening that reaches a peak stress is determined by the work hardening and the work required to rotate and break down the austenite

particles. The flow-softening region derives from the dynamic recrystallization of the austenite phase (14). The type II flow curves of the D1 (TT) and D2 (TT and CR) samples display different features: a greater loading stress and smaller rate of hardening in the work hardening region. On the other hand, the microstructure of these samples showed austenite particles with a Widmansttäten-like structure. It has been reported, for this kind of steel, that austenite obeys the Kurdjumov-Sachs relationship respect to ferrite, being coherent or semi-coherent with the latter (5). This coherency increases the degree of interaction between the precipitates and glide dislocations. At the onset of straining, the austenite particles, therefore, inhibit straining of the softer matrix, increasing the yield stress. As straining proceeds, the elimination and rearrangement of dislocation trigger the process of dynamic recovery. Subgrain formation and misorientation begin to destroy this coherence and the work hardening rate decreases. Moreover, because the austenite particles are not contiguous in these samples, the work of rotating and fragmenting the particles is less and the rate of work hardening and peak stress are decreased, as revealed in Figure 3.

#### Conclusions

The presence of small austenite particles with a Widmansttäten-like structure inside the ferrite matrix changed the shape of flow stress in duplex stainless steels strained at high temperatures. The austenite phase produced by solid-state transformation of the ferrite is coherent with the latter and increased the degree of interaction between the precipitates and glide dislocation, increasing the yield stress. As formation the straining proceeded, misorientation of subgrains began to destroy this coherence, causing a decrease in the work hardening rate and changing the flow curve shapes from type I to type II.

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