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Effect Of Heat Treatment On Microstructure And Mechanical Properties Of Duplex Stainless Steel

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ABSTRACT

Present study concerns the effect of deformation and heat treatment on the microstructure and mechanical properties of a duplex stainless steel. While hot rolling causes the coarse distribution of the constituent phases (ferrite and austenite), 50% cold rolling results into the elongated and splintered two – phase structure. Supersaturated ferrite structure established by water quenching from 1300°C results into the strengthening due to the formation of fine dispersed austenite precipitates within ferrite grain after isothermal heat treatment (1000°C, 0.5 hour). Duplex structure consisting of ferrite and austenite in a fine-grained form is obtained after isothermal heat treatment of cold rolled sample. Cold deformed and heat treated steel exhibits best combination of strength and ductility among all the investigated steel samples.

1. INTRODUCTION

Duplex stainless steels are a separate class of steels intermediate between the ferritic and austenitic stainless steels. Thus the duplex stainless steels combine some of the characteristics of both ferritic and austenitic steels. The strength of the duplex steels is greater than that of the austenitic steels. Thus for some engineering designs the duplex steels offer the optimum materials selection. The high corrosion resistance and the excellent mechanical properties combination of duplex stainless steels can be explained by their chemical composition and balanced ('duplex') microstructure of approximately equivalent volume fractions of ferrite and austenite[1]. High strength and corrosion resistance come from the ferrite, whereas the austenite phase influences ductility and resistance to uniform corrosion[2]. Firstly, the chemical composition based on high contents of Cr and Mo, improves intergranular and pitting corrosion resistance, respectively. Moreover, additions of nitrogen can promote structural hardening by interstitial solid solution mechanism, which raises the yield strength and ultimate strength values without impairing toughness. Secondly, the two-phase microstructure guarantees higher resistance to pitting and stress corrosion cracking in comparison with conventional stainless steels. Duplex stainless steels (DSSs) are suitable alternative to conventional austenitic stainless steels. They are being increasingly used in oil extraction, paper manufacturing, chemical, petrochemical, nuclear and marine industries[3-5].

Nowadays DSS products are obtained by different processes e.g., casting, forging, rolling etc. The microstructure and mechanical properties of the manufactured product strongly depend on the specific process. Present study aims to study the structural and associated mechanical property changes after the deformation and heat treatment. Thermomechanical processing, which combines both deformation and heat treatment is very effective for microstructure control and various treatments have been developed to improve the mechanical properties without sacrifice of corrosion resistance. However, thermomechanical treatment is still lacking in the field of stainless steel as compared to low and high alloyed carbon steels and hence it is attempted for the improvement of mechanical properties of materials. Attempts have also been made to study the effect of cold rolling prior to heat treatment on the investigated stainless steel.

2. EXPERIMENTAL PROCEDURE

The duplex stainless steel was obtained as cast form with approximately 50 mm square cross section. Then the material was homogenised at 1200°C for 120 minutes in a resistance-reheating furnace. The homogenised ingots were forged into bars of 12.5 mm × 12.5 mm section with a reduction ratio of 1:16 using a pneumatic hammer of 1.0-ton capacity. The forged bars were soaked at 1200°C and hot rolled in a laboratory scale two - high rolling mill (10 HP) with 750°C finish rolling temperatures (FRT) down to a thickness of approximately 6 mm in three passes (Fig. 1). The FRT for the steel was fixed at 750°C with an aim to incorporate approximately 10% deformation as was feasible under the experimental facility. After completion of rolling, samples were cooled in air. The chemical composition of the investigated steel is shown in Table 1. The material was heat treated in different schedules as described in Table 2.

To study the microstructures, the samples were subjected to the standard grinding and polishing techniques before etching. The steel sample was etched using aqua regia solution (75% HCl and 25% HNO_3 mixture)[6]. Properly etched samples were examined under the ZEISS (Axiovert 40 MAT) optical microscope at different magnifications and representative photomicrographs were presented. Quantitative metallographic studies were also done using image - analysis technique in order to describe the microstructures. Microstructures have been quantified by the OLYSIA - m3 soft imaging system developed by Olympus.

Table 1
Chemical composition of investigated duplex stainless steel casting (wt%)

C	Mn	Si	S	P	Cr	Ni	Mo	N	Fe
0.03	1.02	0.70	0.002	0.02	21.73	5.15	2.96	0.16	Balance

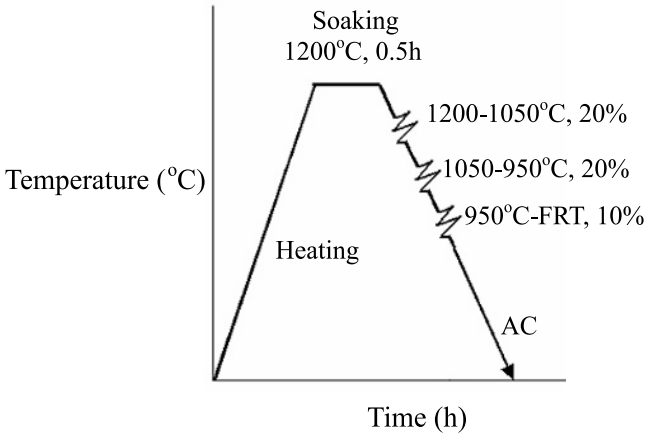

Fig. 1 : Schematic illustration of hot rolling schedule of the investigated steels.

The different phases are identified by color separation and corresponding measurement gives the phase analysis result for each image frame. The mean of the phase analysis result has been reported here.

Brinell - cum - Vickers hardness tester (Model: BV - 250 (SPL)) was used to measure the Brinell hardness of the thermomechanically processed as well as heat treated samples. At least six indentations were taken on each sample and the average hardness values are listed in Table 2. The error in hardness measurement was approximately ± 3% of the recorded Brinell hardness number (BHN).

Room temperature tensile testing was carried out using a computer controlled Instron - 4204 testing machine with a crosshead velocity of 0.5 mm/min. The test specimen was prepared as per ASTM Standard (ASTM: Vol. 03.01: E8M - 96). The yield strength (YS), ultimate tensile strength (UTS), percent total elongation (% TEL) were determined from the machine out put and shown in Table 2. The error in YS and UTS measurement was noted as ~ ± 3% and the same for percent elongation was ~ ± 5%.

3. RESULTS AND DISCUSSION

Table 1 presents the chemical composition of the investigated duplex stainless steel casting obtained after spectroscopic analysis using an Optical Emission Spectrometer (ARL - 2460).

3.1 Microstructural Evolution during Thermomechanical Processing

Present investigated duplex stainless steel essentially contains BCC ferrite (α) and second phase FCC austenite (γ) that is precipitated from α. Figure 2(a) shows that the microstructure of the as-cast duplex stainless steel consists of dispersed γ and continuous α phase. The microstructure shows predominantly γ but contains considerable amounts of α either in the dendritic cores or interdendritic spaces. The γ phase found at the grain boundary contains projections inside α grain. These projections are indicative of Widmanstätten transformation to γ that occurs during solidification.

The $Cr_{equivalent}/Ni_{equivalent}$ ratio obtained through equations (1) and (2) was 1.69.

$$Cr_{eq} = Cr(\%) + 1.5\ Si(\%) + 1.4Mo\ (\%) + Nb\ (\%) - 4.99 \quad (1)^{7}$$

$$Ni_{eq} = Ni(\%) + 30C\ (\%) + 0.5Mn(\%) + 26(N-0.02\%) + 2.77(2)^{7}$$

ASTM standard A800/A800M[7] indicated 46% ferrite for this $Cr_{equivalent}/Ni_{equivalent}$ ratio. Result obtained from image analysis measurement was 44% ferrite and it is a good agreement with A800/A800M. The presence of nitrogen strongly favours the occurrence of austenite in the microstructure.

Table 2
Mechanical properties and volume % of the constituent phases obtained after various treatments for the investigated steel

Treatments	γ/α (%)	Hardness (BHN)	YS (MPa)	UTS (MPa)	TEL (%)
As received cast sample	56/44	229	462	660	29
Hot rolled and air cooled	60/40	252	476	723	39
50% cold rolled sample	60/40	294	814	890	18
Cast sample subjected to standard solution treatment at 1140°C for 0.5 hour followed by furnace cooling to 1000°C and isothermal treatment at 1000°C for 0.5 hour followed by water quenching	46/54	224	456	649	34
50% cold rolled sample subjected to isothermal treatment at 1000°C for 0.5 hour followed by water quenching	45/55	252	498	760	37

The cast structure after hot rolling shows the coarse two – phase (α + γ) structure elongated along the longitudinal direction as shown in Fig. 2(b). During hot rolling γ particles exerted less pinning effect on the migration of α grain boundary[8]. This results into the elongated coarse two - phase structure with few equiaxed γ grains. The measured ferrite content is 40%. The micrograph of the hot rolled sample when subjected to 50% cold deformation is shown in Fig. 2(c). It exhibits finer elongated layers of two – phase (α + γ) structure as compared to that of the hot rolled sample (Figure 2(b) vis-à-vis Fig. 2(c)). The two – phase structure is largely splintered by the cold working.

3.2 Effect of Heat Treatment on Microstructure Formation

Figure 3(a) exhibits the dual phase structure of ferrite (54%) and austenite phase (46%) when the cast steel was solution treated at 1140ºC for 0.5 hour followed by furnace cooling to 1000ºC and isothermal treatment at same temperature for 0.5 hour followed by water quenching. This heat treatment is considered as the standard heat treatment procedure for casting [9,10] and has been attempted here. γ phase precipitates along the coarse α grain boundaries and within α grains. Coarse two-phase (α + γ) structure is formed due to the slow cooling (furnace cooling) of the steel from the solution treatment. The acicular/Widmanstätten austenite present in cast structure transformed to coarse equiaxed type of austenite after heat treatment.

The hot rolled specimen was solution treated at higher temperature (1300°C) in α single-phase region followed by water quenching to obtain a supersaturated α at room temperature and Figure 3(b) displays the micrograph of the same. α matrix grains are huge since the α grain size is determined by the solution treatment at higher temperature. On quenching from 1300°C into water it is found impossible to retain wholly ferritic structure. The formation of γ precipitate was unavoidable. The measured ferrite content is 97% and the rest 3% is austenite precipitated along the ferrite grain boundary and triple points. Figure 3(c) shows the micrograph obtained after isothermal treatment (1000°C, 0.5 hour) of the super saturated predominantly α matrix structure. Precipitation of lighter γ phase takes place from supersaturated α phase and was visible at the grain boundaries and densely precipitated within the ferrite grain. There was some additional grain growth of those few austenite particles which were present after water quenching from above 1300°C. γ precipitates appear to be both equiaxed and elongated morphologies. The measured austenite content was 35%.

Figure 3(d) shows the mixture of fine, coarse granular and elongated nature of precipitation of austenite phase within the ferrite phase when the 50% cold deformed steel was isothermally treated at 1000ºC for 0.5 hour followed by water quenching. With this treatment fine γ particles are uniformly precipitated. Elongated γ layers in the cold rolled specimen turns to a row of roughly equiaxed γ grains separated from each other. Some authors[11] have reported that the phase boundaries play the key role in the plastic deformation. The elongated austenite grains show the signature of cold rolling operation, which was not recovered fully after isothermal treatment. During heat treatment the two metallurgical processes i.e., recovery or recrystallisation of the α phase and precipitation of the second phase (γ) occur simultaneously. At the early stage of heat treatment it is believed that recovery of α phase occurs rapidly and sub-grain structure of α phase is formed prior to the precipitation of γ[12]. Then γ phase precipitates at a sub-grain boundaries as shown in Fig. 3(e). The measured volume percent of γ was 45%.

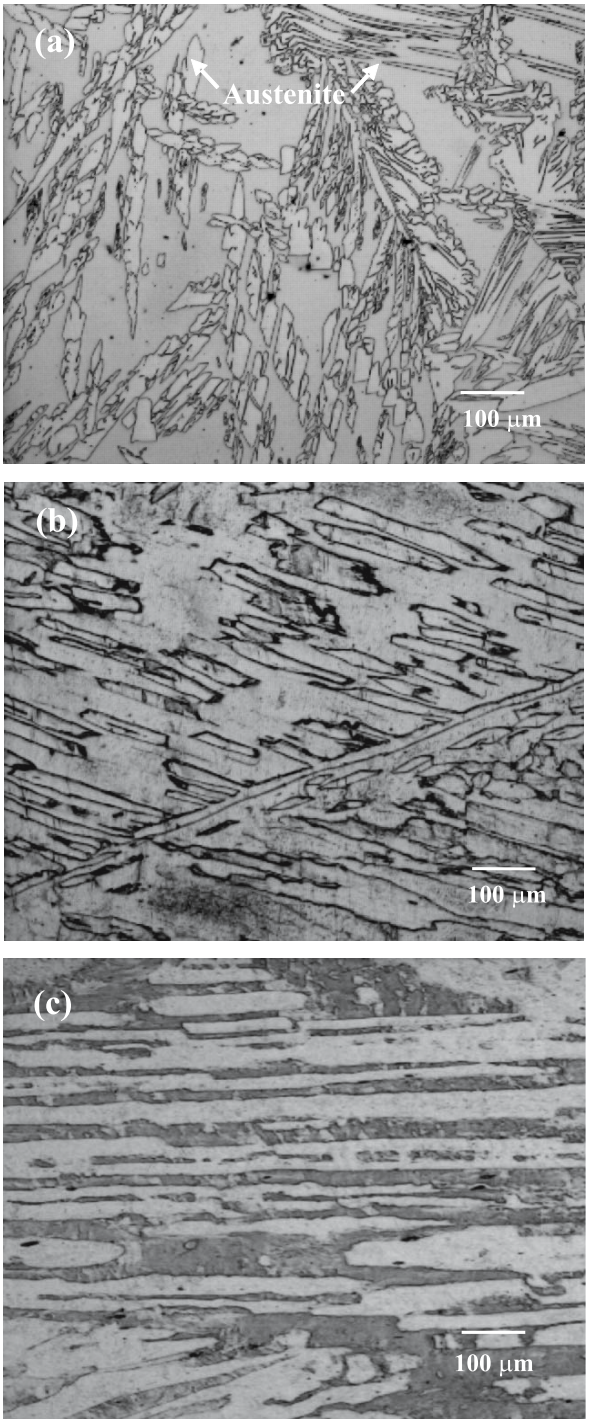

Fig. 2 : Optical micrographs (a) cast steel showing acicular/ Widmanstätten austenite phase with considerable amounts of ferrite either in the dendritic cores or interdendritic spaces, (b) coarse two – phase (austenite + ferrite) structure obtained after hot rolling and (c) 50% cold deformed sample showing finer, elongated and splintered two – phase structure.

3.3 Effect of Thermomechanical Processing and Heat Treatment on Mechanical Properties

3.3.1 Hardness Measurement

Table 2 shows that the hardness value of cast duplex stainless sample is 229 BHN. The hardness value increases when the cast sample was subjected after hot rolling and air cooling. Hot rolling causes 50% reduction in thickness of the cast sample and the increased percentage and distribution of γ phase within α causes the 10% improvement in hardness (252 BHN). Further 17% improvement in hardness (294 BHN) is recorded when the hot rolled sample was subjected to 50% cold rolling. As for other metallic materials 50% cold working further increases the hardness because of the work hardening behaviour of the constituent phases.

The cast structure when subjected to standard solution treatment (1140°C, 0.5 hour) and subsequent water quenching exhibits lower (224 BHN) hardness than that of the cast sample. The reduced percentage of γ (46%) as compared to cast sample (56%) and coarse equiaxed austenite dispersion within ferrite results in lower hardness of the solution treated sample (Fig. 3(a)). Isothermal heat treatment at 1000°C of the

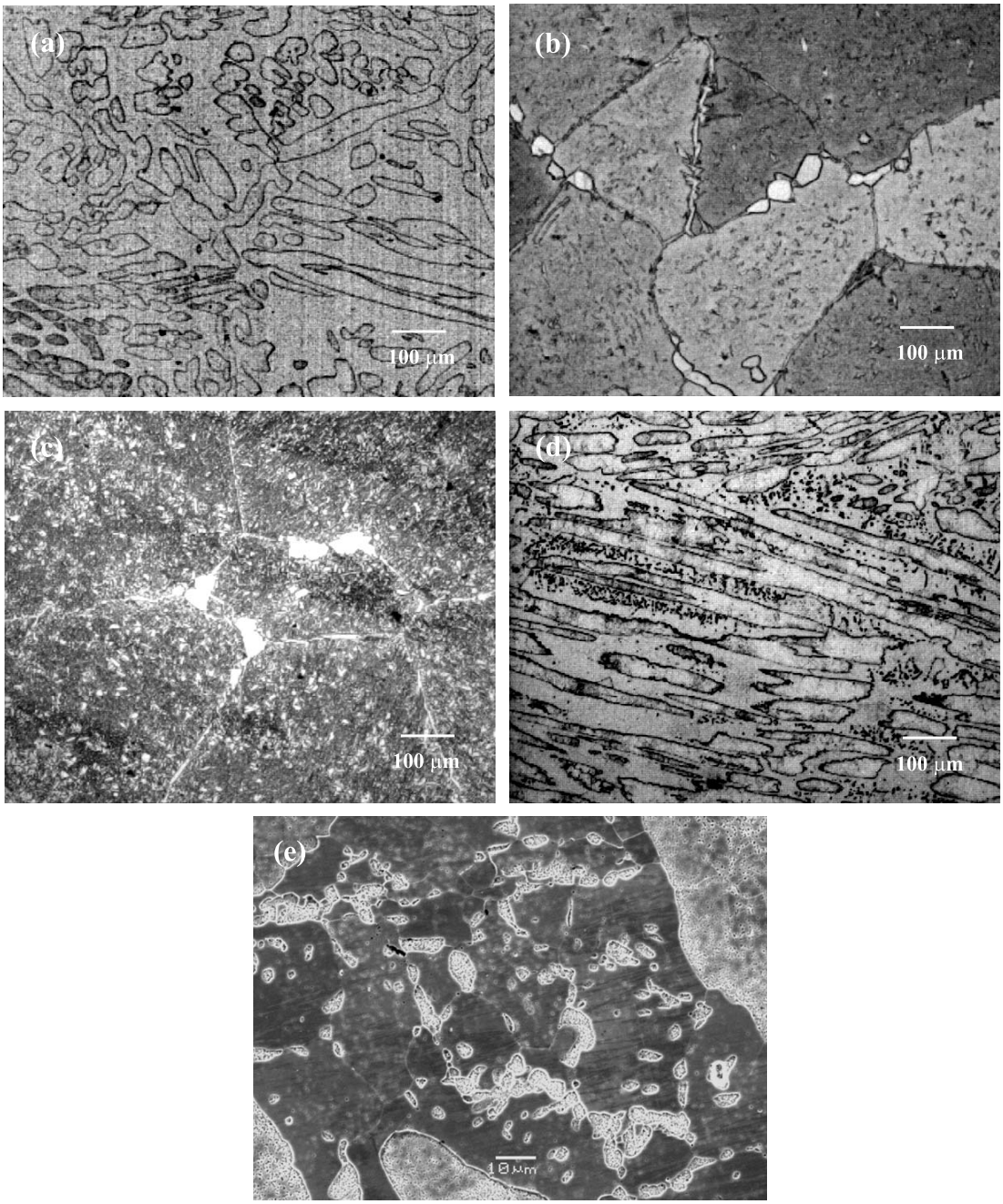

Fig. 3 : Optical micrograph of (a) cast steel after standard solution treatment showing the equiaxed type of dispersed austenite within continuous ferrite phase, (b) water quenching from 1300°C results in predominantly coarse ferrite grain, (c) isothermal treatment (1000°C, 0.5 hour) of supersaturated ferrite results into the fine dispersion of austenite phase, (d) elongated austenite layer in the cold rolled specimen turns to a row of equiaxed austenite grain after isothermal treatment and (e) scanning electron micrograph of the cold deformed and heat treated sample showing the austenite precipitates at ferrite sub-grain boundaries.

cold rolled sample generally causes the drop of hardness values (252 BHN). This may be explained that in agreement with the earlier research the heat treatment at 1000°C causes the recovery of the deformed structure[13, 14] which in turn leads to drop of hardness value.

3.3.2 Tensile Properties

Table 2 shows that tensile properties of the investigated duplex stainless steel samples under various conditions. The major characteristic is their high yield strength (462 – 498 MPa). The ultimate tensile strength is also high (660 – 890 MPa) while the elongation is generally greater than 25% except for the cold rolled sample. Cast structure records the lowest yield strength (462 MPa) and tensile strength (660 MPa) and poor elongation value (29%) because of the chemical segregation as well as presence of acicular/ Widmanstätten type of dispersed austenite phase at the segregated regions (Fig. 2(a)). Hot rolled sample records higher yield (476 MPa), tensile strength (723 MPa) and elongation (39%) value as compared to those of the cast sample. The higher austenite/ferrite ratio and mixture of equiaxed and elongated austenite phase with coarse ferrite (Fig. 2(b)) may be hold responsible for the improved tensile properties of the hot rolled sample. Maximum strength 890 MPa is achieved in case of cold rolled sample because of the work hardening behaviour of the constituent phases in spite of having similar austenite/ferrite ratio to that of the hot rolled sample.

On the other hand, solution treatment (1140°C) followed by isothermal treatment at 1000°C and rapid cooling (water quenching) causes the drop of yield (456 MPa) and tensile strength (649 MPa) and improvement of elongation (35%) of the cast sample. This can be explained by the lower austenite/ ferrite ratio and coarse and uniform dispersion of austenite phase within ferrite phase (Fig. 3(a)) 50% cold rolled sample when subjected to isothermal treatment at 1000°C increases yield strength (498 MPa) and tensile strength (760 MPa) over the hot rolled sample. The pronounced elongation of the hot rolled sample which was largely destroyed by cold working (18% total elongation) can be recovered (37%) by this isothermal treatment of the cold deformed sample. These high mechanical properties result from accumulated contribution of elongated, equiaxed austenite phase, recovered deformed structure and presence of fine γ phase precipitates at the α sub-grain boundaries (Fig. 3(e)) in spite of having lower austenite/ferrite ratio.

4. CONCLUSIONS

- Cast structure results into the precipitation of dispersed Widmanstätten austenite within continuous ferrite phase.
- Hot rolling causes the coarse distribution of the constituent phases (α + γ) in the investigated steel. The two-phase structure become elongated and splintered by the subsequent 50% cold deformation.
- Duplex structure consisting of ferrite and austenite in a fine-grained form is achieved in cold rolled sample after isothermal heat treatment at 1000°C for 0.5 hour.
- Cold rolled and isothermally heat treated steel exhibits best combination of strength and ductility among all the investigated steel samples.
- Water quenching from 1300°C results into the formation of coarse α grained structure with little amount of γ forming at the grain boundaries during cooling which in turn results in the dense precipitation of fine γ phase inside α grain after isothermal heat treatment.

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