

# Effects of solution treatment on microstructure and mechanical properties of 316 L and 430 L Duplex Stainless Steels sintered in different atmospheres

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## ABSTRACT

In this research, the effects of solution annealing temperature on Duplex stainless steels (DSS) were investigated through Powder metallurgy (P/M) route. The steel samples studied in two different compositions sintered in two different atmospheres such as hydrogen and partial vacuum. The sintered DSS samples were solution annealed at 1250°C for one hour and followed by water quench to analyze the phase structures of both ferrite and austenite during solution treatment and to study the effect of solution treatment on mechanical properties. Density of solution treated DSS have slight increase than the sintered steels. The yield strength and tensile strength of solution treated DSS exhibited higher values than the sintered one. The significant increase in properties of solution treated DSS were influenced by density, chemical compositions and sintering atmospheres.

**KEY WORDS:** Duplex Stainless Steel, Hydrogen, partial vacuum, Solution Anneal, Microstructure, Ferrite content.

## 1. INTRODUCTION

Duplex stainless steels (DSS) officiate as superior one with combined advantageous of (non-hardened type by heat treatment) ferritic and austenitic phases. DSS includes the combined properties of the higher strength and stress corrosion resistance of ferritic steels and greater ductility, toughness and corrosion resistance of austenitic steels. (Martins, 2005; Kashiwar, 2012; De Lacerda, 2015). The novel applications of duplex stainless steels are high-pressure vessels, high pressure piping (higher strength and cost saving over austenitics), temperature sensors, and includes its cardinal applications in nuclear, petrochemical and marine industries (Ghosh and Mondal, 2008)

Heat treatment is the significant role in DSS which exhibit the materials become thermodynamically metastable structure since the greater accumulation of solute atoms in solid solution Marcelo (Martins, 2005) Several researchers reported that the solution annealing heat treatment which persuades the microstructure become balanced phases of austenite and ferrite without any intermetallics. Solution treatment is solitary route and very eminent measures to sustain the high localized and pitting corrosion resistance because of volumetric equilibrium fraction of ferrite and austenite phases in DSS and obtaining passivation effect by homogeneous distribution of alloy elements. Solution treatment which makes multifarious on the DSS from original phase constituents by increasing the percentage of alloy contents like Cr, Mo and Ni in both phases due to decreased intermetallic phases. For solution annealing, many researchers given their conclusions on different annealing temperature, the  $\sigma$ -phase can be annihilated by the temperature above 1050° C -1250 °C Marcelo (Martins, 2005; Yang , 2011) but facilitate an intergranular secondary austenite ( $\gamma_2$ ) precipitations on DSSs Yang , 2011). Due to high cooling rate of 30 - 40 °C/s, the  $\sigma$  phase can entirely eliminated on heat treatment at 1300°C (De Lacerda, 2015) however the strong recrystallization and grain becomes coarsening of both phases about 1200 °C but the higher solution treatment temperature of 1300°C shows multifarious morphology with rich in ferrite grain size and the appearance of acicular austenite grains on boundaries of ferrite grains. The proliferation in solution treatment temperature which cause the levitate in the volume fraction of ferrite (Jianquan Wan, 2014; Yang, 2011). The ferrite fraction can be increased by the solution heat treatment at 1050 °C is 42 - 48.9% and increased to 52 - 55% and maximum of 69% for sample solution annealed at 1150 °C and 1250 °C respectively (Naghizadeh, 2015; Yang , 2011).

In the present investigation, type 316 L has exploited because of its more corrosion resistance than conventional 304 / 304 L type and as per the many researcher's opinion the corrosion resistance and strength are very important properties in particularly for stainless steel families due to its unavoidable use in highly corrosive and high strength necessity applications. The main objective of this work is to improve the mechanical properties of P/M DSS by treating solution annealing at 1250°C for 1 hour followed by water quenching of sintered DSS developed from pre alloyed powders and elemental powders. Densification properties, micro-structural Analysis and mechanical properties evaluations were conducted on the sintered and solution treated DSS to observe the favor and unfavor effect of solution annealing heat treatment.

## 2. EXPERIMENTAL

In this paper, sintered AISI 316L and AISI 430L Duplex stainless steel (DSS) samples were taken for the investigation. The sintered DSS samples were solution annealed at 1250°C in electrical muffle furnace for one hour

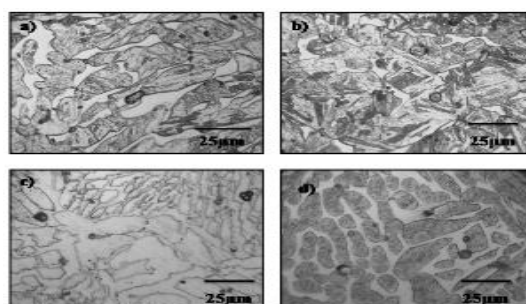
and followed by water quench to analyze the phase structures of both ferrite and austenite during solution treatment. The DSS samples having two different compositions prepared from the equal weight combination (50 wt %) of ferritic and austenitic powders and the other was prepared from elemental addition of chromium, nickel and molybdenum in proper ratio to obtain the duplex structure. The elemental concentrations of two different DSS compositions are shown in the table 1. The sintered steels have the dimensions of 30 mm diameter and 12 mm height were sintered at  $1350 \pm 10^\circ\text{C}$  for 2 hrs in two different atmospheres such as hydrogen and partial vacuum. During Sintering in hydrogen atmosphere, 250 ml of the gas per minute was passed into the sintering zone during partial vacuum sintering the pressure level was maintained at  $10^{-1}$  mbar and hydrogen was used as the back filling gas. Sintered densities and solution treated densities were measured from mass and physical dimensions. The metallographic samples of sintered and solution treated DSSs were polished and etched with Berahaa reagent. The microstructure was observed in an optical microscope (Metatec, India) using an Image Analyzer software. Tensile tests were performed on a digital tensometer (M/s. Kundale India Ltd. Bangalore) with a strain rate of 1 mm/min. Micro tensile samples as per the ASTM E8 standard were machined and tensile strength was evaluated. Hardness of the samples were performed on a Rockwell Hardness Tester (FIE Model) in A scale (diamond indenter and 60 Kg major load) was used throughout the investigation. SEM micrograph and Energy Dispersive Spectroscopy (EDS) analysis of the solution treated DSS were evaluated using scanning electron microscope (Hitachi Model S3000HI).

**Table.1. Chemical composition of Duplex Stainless Steels**

Composition	Elemental Concentration (%wt)								
	Cr	C	Ni	Si	Mn	Mo	Fe	Cr <sub>eq</sub>	Ni <sub>eq</sub>
DSS A	16.58	0.018	6.22	1.05	0.10	1.10	Bal	19.26	6.81
DSS B	18.93	0.016	8.59	0.95	0.09	3.99	Bal	24.35	9.12

### 3. RESULTS AND DISCUSSION

**Micro-structural Analysis:** AISI 316L and AISI 430L Duplex stainless steel (DSS) sintered samples were solution annealed at  $1250^\circ\text{C}$  and followed by water quenching. It is necessary to study the effect of solution treatment on the phase microstructures are shown in figure. 1. The optical micrographs in the figure. 1(a) & 1(b) and 1(c) & 1(d) are the microstructures of solution treated DSSs sintered in hydrogen atmosphere and partial vacuum atmosphere respectively.

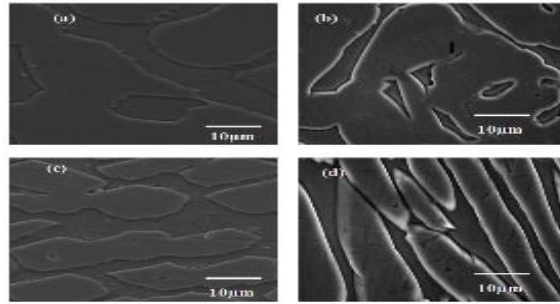


**Fig.1. Optical micrographs of Duplex stainless steels Solution treated at  $1250^\circ\text{C}$  (a) Solution treated DSS A sintered in hydrogen atmosphere (b) Solution treated DSS B sintered in hydrogen atmosphere (c) Solution treated DSS A sintered in partial vacuum atmosphere (d) Solution treated DSS B sintered in partial vacuum atmosphere**

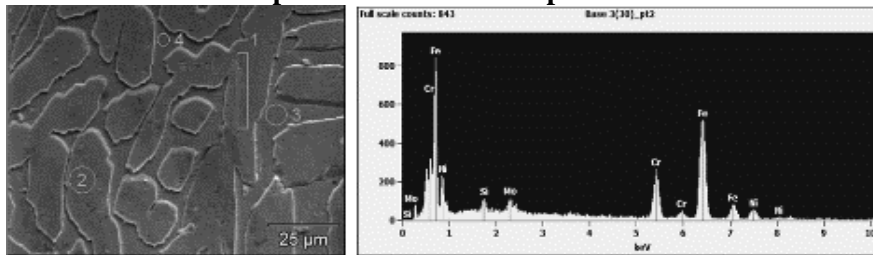
The microstructures shown in figure.1 (a) and (b) consist of duplex structure containing austenitic grain which appears as light in colour and surrounded by the dark/grey ferrite matrix (Naghizadeh, 2015; De Lacerda, 2015) The large ferrite grains with allotriomorphic austenite along the grain boundaries are observed in the microstructure and the structure is termed as Widmanstatten austenite.

The fig. 1 (c) and (d) shows homogeneous distribution of ferrite and austenite phases in the solution treated DSSs sintered in partial vacuum conditions. The higher solution heat treatment leads to a strong recrystallization and grain coarsening of both phases (De Lacerda, 2015). From the microstructures shown in fig.1(a) and 1(c), no precipitates are observed in ferrite and at the interfaces of ferrite and austenite grains of the solution annealed DSS A (equal wt% of both ferrite & austenite) composition sintered in both conditions. Moreover in the composition DSSB, larger volume of ferrite was observed than the DSS A. An increase in chromium equivalent number in the DSS B.

Composition enhances ferrite volume in the phase structure. This trend has been replicated for both hydrogen and partial vacuum sintered conditions. Solution annealing treatment at  $1250^\circ\text{C}$  slightly enhanced the ferrite content in the duplex microstructure due to the dissolution of austenite in to ferrite matrix (Yang, 2011; Chen Hong, 2011). From the microstructure it is cleared that fig 1(a) and 1(c) have less volume of ferrite content than its counter parts. Figure 1(d) shows that the microstructure with clear region of austenite phase, but small dark precipitation like appearance was observed inside the ferrite phase grain only.

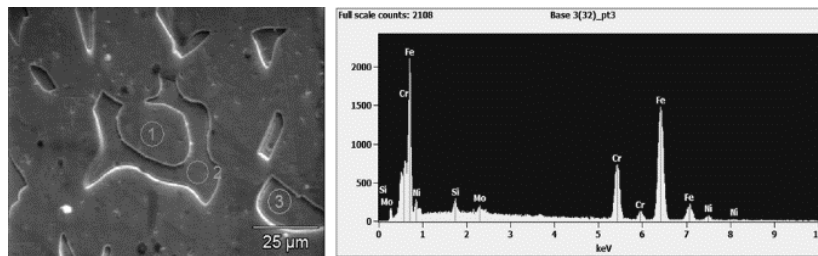


**Fig.2. SEM micrographs of Duplex stainless steels Solution treated at 1250°C (a) Solution treated DSS A sintered in hydrogen atmosphere (b) Solution treated DSS B sintered in hydrogen atmosphere (c) Solution treated DSS A sintered in partial vacuum atmosphere (d) Solution treated DSS B sintered in partial vacuum atmosphere**



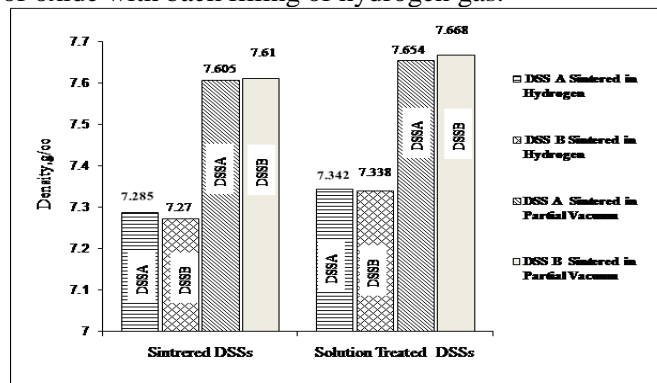
**Fig.3. SEM - EDS analysis on the defined grain (Ferrite) of solution treated DSS B sintered in partial vacuum**

The figure. 2 shows the SEM-micrographs of solution treated DSS sintered in hydrogen (fig.2.a & 2b) and partial vacuum (fig.2c & 2d) conditions. The figure.3 shows that SEM – EDS analysis of ferrite grain and at the interface of austenite & ferrite region of solution treated composition DSS B sintered in partial vacuum. No evident of chromium rich precipitates was observed in ferrite grains and at the interfaces, instead of that, the stabilizing elements such as chromium, silicon and molybdenum are found during EDS analysis. Similarly the composition DSS A does not have any precipitates in the ferrite grain. This trend has been observed for DSS B (fig.4) sintered in hydrogen atmosphere, it has been confirmed through SEM-EDS report.



**Fig.4. SEM - EDS analysis on the defined grain (austenite) of solution treated DSS B sintered in Hydrogen**

**Density of solution treated DSSs:** The figure.5 shows the densities of different duplex stainless steels both in sintered & solution treated Conditions. Vacuum sintered stainless steels having better densities after solution treatment than the hydrogen sintered steels. The reason is that the diffusional kinetics in partial vacuum steels is better due to more reduction of oxide with back filling of hydrogen gas.



**Fig.5. Densities of different duplex stainless steels both sintered & Solution treated Conditions**

In the figure.5, it is observed that the solution annealing at 1250°C, which makes further improvement on the density of steels when compared to sintered conditions. The significant effect of solution treatment temperature was proved by Naghizadeh (2015).

**Mechanical Properties of Solution Treated DSSs:** The mechanical properties of sintered and solution treated duplex stainless steels are drawn as bar graphs in the figure.6 and figure.7 respectively. The figure.6 (b) and 7(b) have shown the tensile bar graphs of different DSS sintered (partial vacuum) and solution treated conditions respectively; the higher tensile strength of 824 MPa is observed in the solution treated DSS A sintered in partial vacuum atmosphere. The increased tensile strength of 8- 9 percent has been observed with the influence of solution treatment temperature which makes more density and it is evident from the bar graph (fig.5). But the DSS A (solution treated condition of partial vacuum steel) has lesser yield strength of 438 MPa than DSS B of 444 MPa. The increased yield strength in DSS B is due to more ferrite volume and it is enhanced the chromium and nickel equivalent number in that particular composition. Less ferrite content in the duplex composition DSS A decreases yield strength. Similarly more volume of austenite in the duplex composition increases the ultimate tensile strength and elongation. Hence, DSS A attained higher tensile strength and more toughness.

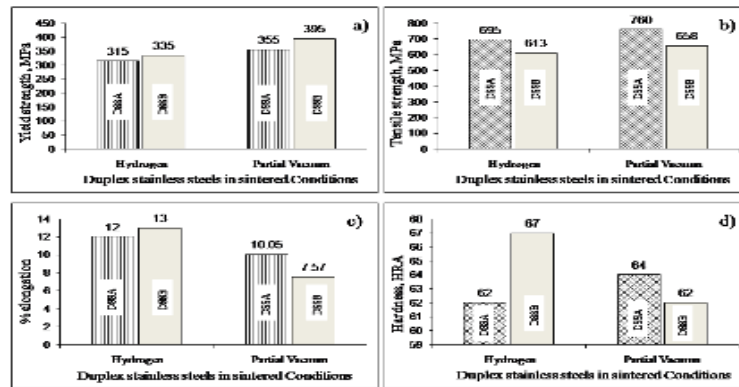


Fig.6. Mechanical properties of DSS sintered in hydrogen & Partial vacuum atmosphere

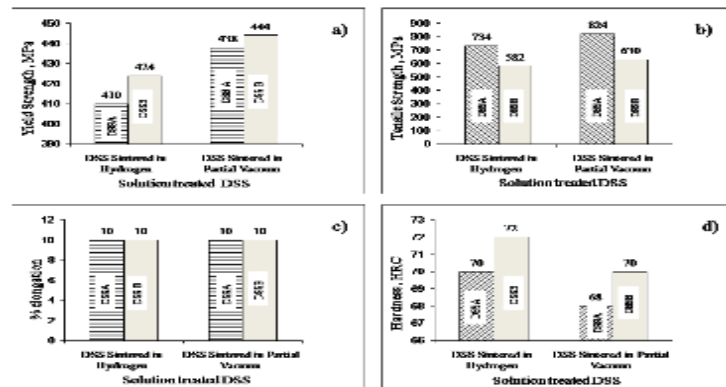


Fig.7. Mechanical properties of 1250°C Solution treated DSS sintered in hydrogen & Partial vacuum atmosphere

After solution treatment, the ferrite volume has been increased due to the dissolution of austenite in to ferrite matrix. The larger amount of ferrite is the reason for that hardness values are improved due to solution annealing treatment at 1250°C.

#### 4. CONCLUSION

An investigation deals the effects of solution annealing at 1250° C on the microstructure and mechanical properties of AISI 316L and AISI 430L Duplex stainless steel (DSS) reached the following conclusions:

- ❖ SEM – micrograph clearly says that there are no precipitates observed at the ferrite region and at the interface of austenite and ferrite and SEM- EDS analysis gives evident for the precipitation free structures.
- ❖ The solution annealing at 1250° C, which makes further improvement on the density of duplex stainless steels when compared to the sintered steels
- ❖ The higher tensile strength of 824 MPa was observed in the solution treated DSS A sintered in partial vacuum atmosphere. The tensile strength has increased to 8- 9 percent due to the increased density after solution treatment.
- ❖ The increased yield strength and hardness due to increased ferrite volume observed in DSS B but less ferrite content in the duplex composition DSS A decreases yield strength.

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