U.P.B. Sci. Bull., Series B, Vol. 83, Iss. 3, 2021 ISSN 1454-2331

INFLUENCE OF SOLUTION TREATMENT TEMPERATURE ON MICROSTRUCTURAL AND MECHANICAL PROPERTIES OF HOT ROLLED UNS S32760 / F55 SUPER DUPLEX STAINLESS STEEL (SDSS)

Saleh Sabah ALTURAIHI [1]*, Nicolae SERBAN[1], Ion CINCA[1], Mariana Lucia ANGELESCU[1], Irina Varvara BALKAN[1]

The effects of solution treatment temperature on the microstructural evolution and mechanical properties of hot rolled F55 super duplex stainless steel has been studied in this paper. The F55 alloy was hot deformed at 1000°C, with 60% total deformation degree in 5 rolling steps, and heat treated by solution treating at 1000°C, 1050°C and 1100°C for 20 Minutes. The samples microstructure was investigated by Scanning Electron Microscopy-Electron Backscatter Diffraction (SEM-EBSD) techniques. Microstructural characteristics such as weight fraction, distribution and morphology of constituent phases and grain misorientation were analyzed in relation to thermomechanical processing conditions. All samples were tensile tested to quantify the effect of thermomechanical processing on mechanical properties (ultimate tensile strength, yield strength and elongation to fracture). Experimental results showed that the constituent phases in the SDSS alloy can be ferrite phase (δ), austenite phase (γ) and sigma phase (σ), depending on applied solution treatment. Obtained microstructure after hot deformation and solution treatment at 1000°C showed a mixture of ferrite, austenite, and sigma phases. In the case of solution treatments at temperatures above 1050°C the deleterious sigma phase is no longer observed. Investigations on mechanical properties showed that increasing solution treatment temperature, increases elongation to fracture, while strength properties are decreased, all due to the presence / absence of deleterious sigma phase.

Keywords: Super duplex stainless steel (SDSS), hot rolling, solution treatment, microstructure, mechanical properties

1. Introduction

The first-generation Duplex stainless steels were developed more than 70 years ago in Sweden for use in the sulfite paper industry. Duplex alloys were originally created to combat corrosion problems caused by chloride-bearing cooling waters and other aggressive chemical process fluids. The alloy called Duplex because of its mixed microstructure with about equal proportions of ferrite and austenite, Super-Duplex steel provides outstanding resistance to acids, acid chlorides, caustic solutions and other environments in the chemical/petrochemical,

[1] Materials Science and Engineering Faculty, University POLITEHNICA of Bucharest, Romania
*Corresponding author: salehsabah2016@gmail.com

pulp and paper industries [1]. The ‘‘Duplex’’ microstructure is obtained by controlled alloying with nitrogen, nickel and chromium. Small percentages of Mo, Mn, Si, and other alloying elements are also present in these alloys [2]. A less favorable aspect is the poor hot ductility of SDSS, which makes their hot working to be very difficult [1,3,4]. Besides chemical composition, thermo-mechanical treatments have a significant influence on the properties of SDSS, hence the importance of studying this subject to improve their behavior during industrial processing and exploitation. It has been shown that the high alloying content in these steels increases the risk of precipitation of intermetallic phases, with a negative effect on the corrosion resistance and ductility [4–6]. The microstructural evolution of SDSS after hot deformation and subsequent solution annealing is very important for preventing the formation of deleterious intermetallic phases and, therefore, the easiest solution to achieve the desired properties seems to be a proper control of thermo-mechanical treatment [7–11]. The solution annealing improves the mechanical and corrosion characteristics of SDSS by dissolving the secondary deleterious phases at high temperatures, but also the annealing temperature can affect the ferrite phase (δ) and austenite phase (γ) phases proportion, modifying this way the alloying elements partitioning in the two phases (austenite phase (γ) being enriched in Cr and Mo and ferrite phase (δ) in N) and consequently the corrosion resistance of each phase [12, 13]. In this regard, studying the correlation between the solution treatment parameters and microstructural characteristics, mechanical properties can be very helpful [14]. The present work aims to study the microstructural evolution during a brief solution heat treatment applied to a UNS S32760/F55 SDSS alloy, which was previously hot-rolled, with the purpose of improving the hot rolling process and the quality of hot-rolled products. By varying the solution treatment temperature (1000-1050-1100) °C, several microstructural states were obtained. The holding time was 20 minutes. The component phases in different conditions of thermo-mechanical processing were identified and characterized by means of scanning Electron Microscopy-Electron Backscatter Diffraction (SEM-EBSD) techniques, to establish the influence of temperature and duration solution annealing on microstructural and mechanical properties.

2. Materials and method

As-received Super Duplex Stainless Steel UNS S32760 F55 was used in this experimental study, and the chemical composition of the super duplex stainless steel (SDSS) is given in Table 1.

Table 1

Chemical composition of material chosen for study SDSS F55

Element	Fe	Cr	Ni	Mo	Tn	Si	Mn	Cu	Vn
Weight percent %	61.25	27.33	6.78	2.71	0.32	0.49	0.48	0.50	0.14

The samples were heated by caloris srl. furnace, 2 minutes per millimeter and hot rolled by laboratory laminator at 1000°C, the total deformation degree is 60% according to the change in thickness to the original thickness, it was done by five passes of hot-rolling, one millimeter for every pass and the samples were reheated before the next pass of hot-rolling. By using Metkon Micracut 200, the hot rolled sample was cut in to 3 pieces for the solution treatment. Solution treatment was applied on the samples at different temperature (1000-1050-1100) °C in duration 20 minutes by using Naberthrem furnace followed by quenching in water, see Fig. 1. After the heat treatment, the microstructure was investigated by SEM-BSE and SEM-EBSD techniques, with the purpose of observing the microstructural changes produced during the solution treatment. The SEM microscope—Tescan Vega II-XMU SEM (Tescan, Brno, Czech Republic) was fitted with a Bruker Quantax e-Flash EBSD detector. The samples were prepared with a very good surface finish using a precision cutter Metkon servocut-M300. Subsequently, all specimens were hot mounted using a Buehler Simplimet mounting press (Phenocure black phenolic resin; Buehler, Lake Bluff, IL facility) within the cylindrical sampler. All samples were subjected to grinding and polishing using a Metkon Digiprep Accura (advanced high-end grinding and polishing system). Tensile test was done by Instron 3382 machine. To observe the effect of temperature of the solution treatment.

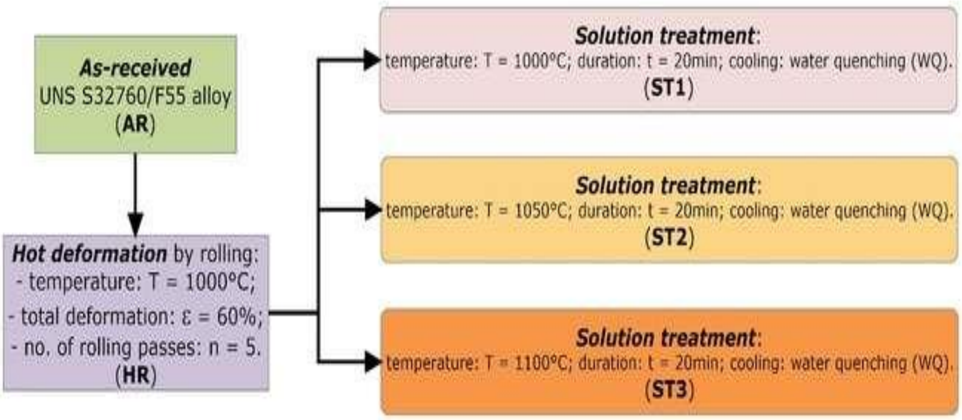

Fig. 1 – Schematic representation of applied thermomechanical processing route.

3. Results and discussion

3.1. SEM-EBSD analysis

The microstructure of SDSS alloys as received contain a mixture of primary phases, with equal percent (50:50) of ferrite phase (δ) and austenite phase (γ), as shown in Fig. 2.

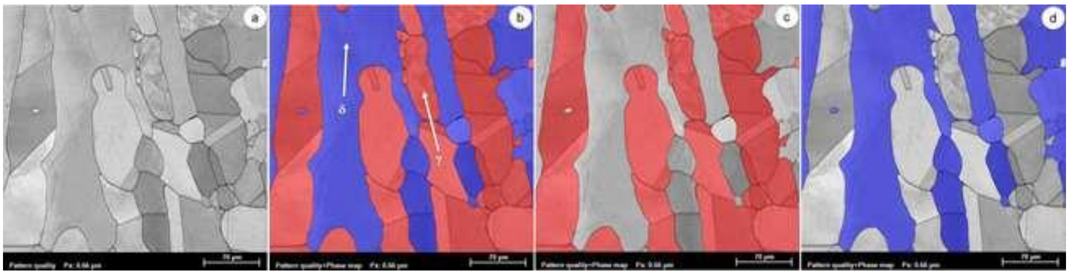

Fig.2. SEM microstructure analysis of sample as received; a) The grains appearance, b) distribution of the phases, c) austenite phase deformation, d) ferrite phase deformation.

The SEM analysis of the hot rolled sample, it was observed that there is an increasing of the volume fraction of austenite phase to (59.7%), decreasing of ferrite volume fraction phase to (36.7%). Due the hot deformation, the undesirable sigma Fe-Cr phase was appeared on the grain boundaries in the with volume fraction (3.55%) which has effect on the mechanical properties. The observed sigma phase was stressed and un-crystallized, as shown in Fig. 3.

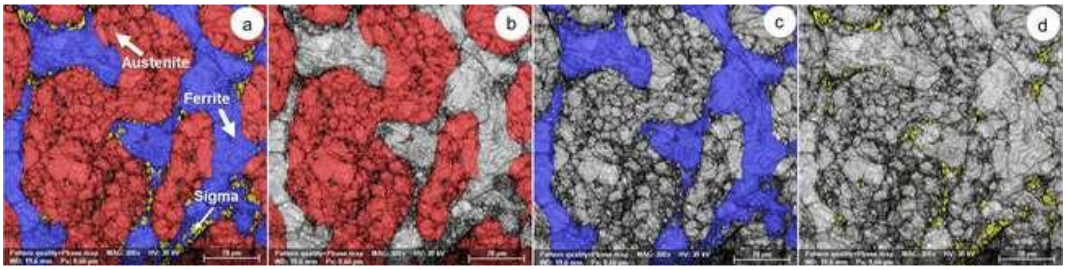

Fig.3. SEM microstructure analysis of sample hot rolled 1.1, a) distribution of the phases, b) austenite phase deformation, c) ferrite phase deformation d) sigma phase deformation.

Fig. 4 shows the Module Orientation average, which is explaining the straining of crystal level inside the grains. It was observed that the ferrite grains disoriented with high straining level more than austenite and sigma phase (Fig. 3 c). The blue color area in the crystal indicants that the crystal orientation is exactly in the average which is mean no straining grains. While the red and yellow have a high straining level of the grain with misorientation angle 23º.

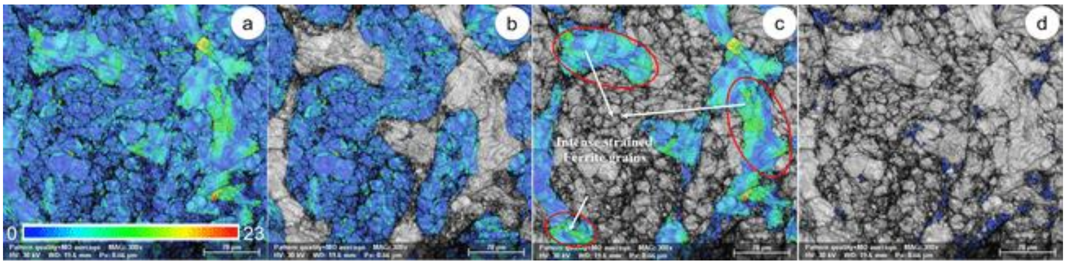

Fig.4 a) MO of the hot rolled sample with maximum value of misorientation angle, b) MO for austenite phase, c) MO for ferrite phase, d) MO for sigma phase.

The solution treatment was applied on the samples. The sample was heat treated, at temperature 1000 °C for duration 20 minutes, the Fig. 5 shows SEM analysis for the samples, it can observe that there is an increasing volume fraction of austenite phase to (62.3%), decreasing of ferrite phase volume fraction to (34.2%) and decreasing volume fraction of sigma phase to (3.5%). While the grains of austenite and ferrite become crystallized and bigger than hot rolled sample as well as generating secondary austenite in the microstructure.

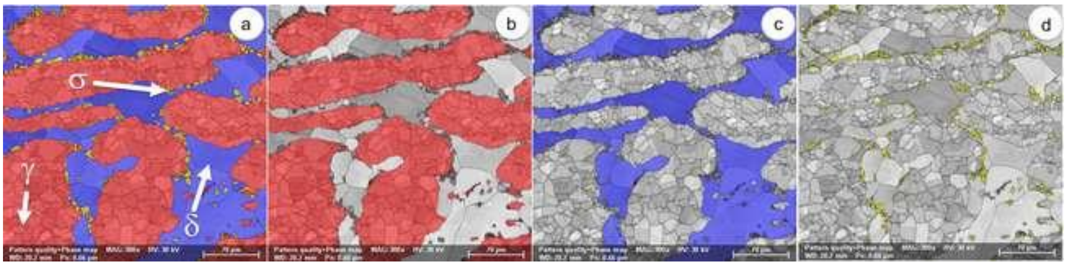

Fig.5. SEM microstructure analysis of sample heat treated 1000°C; a) distribution of the phases, b) austenite phase deformation, c) ferrite phase deformation d) sigma phase deformation.

The misorientation average (MO) of heat treated sample at 1000°C, the misorientation angle decreased to 1.3º and, the crystals were less strained inside the grains, see Fig. 6.

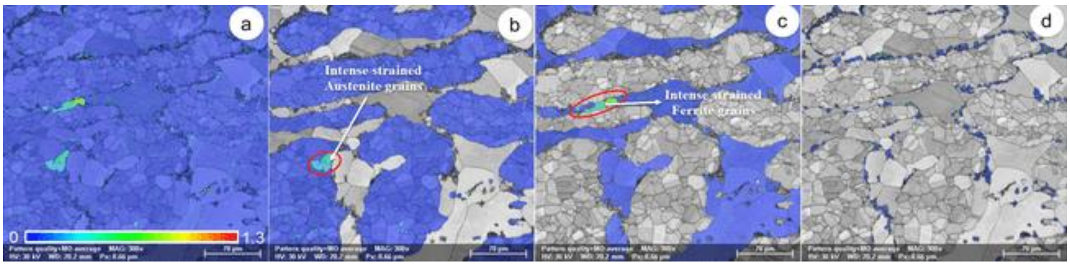

Fig.6a) MO of the sample heat treated at 1000°C with maximum value of misorientation angle, b) MO for austenite phase, c) MO for ferrite phase, d) MO for sigma phase.

To observe the effect of solution treatment temperature on microstructure, the temperature increased to 1050°C at the same duration which is 20 minutes, and microstructure was analyzed. It can be observed that the sigma phase is disappeared in microstructure because of heat treatment temperature more than 1000°C [15], only elongated austenite volume fraction grains decreased to (51.5%) and ferrite volume fraction increased to (49.5%) with bigger grains, also shows there is no more secondary austenite by increasing the temperature of solution treatment. see Fig. 7.

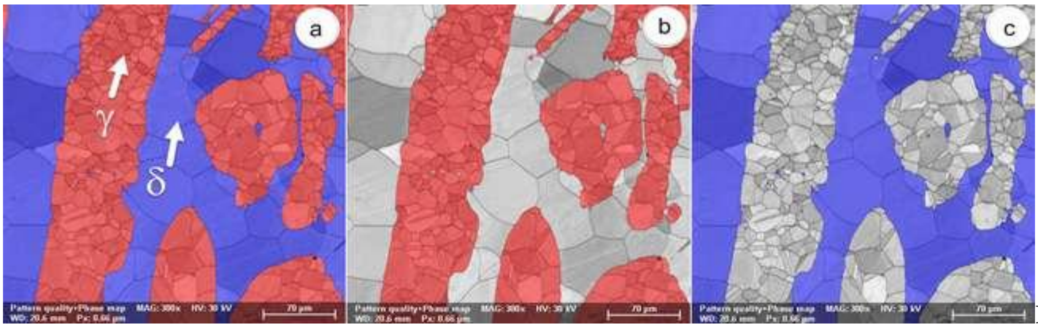

Fig.7. SEM microstructure analysis of sample heat treated at 1050°C, a) distribution of the phases, b) austenite phase deformation, c) ferrite phase deformation

The MO average of heat treatment sample at 1050°C, shows that the misorientation angle decreased to 1.22°, which is less than misorientation angle of hot rolled sample and less than sample which had solution treatment at 1000°C in the same duration. The crystals strained level inside the grains was decreased, except some grains in hot rolled sample in comparison, as shown Fig. 8.

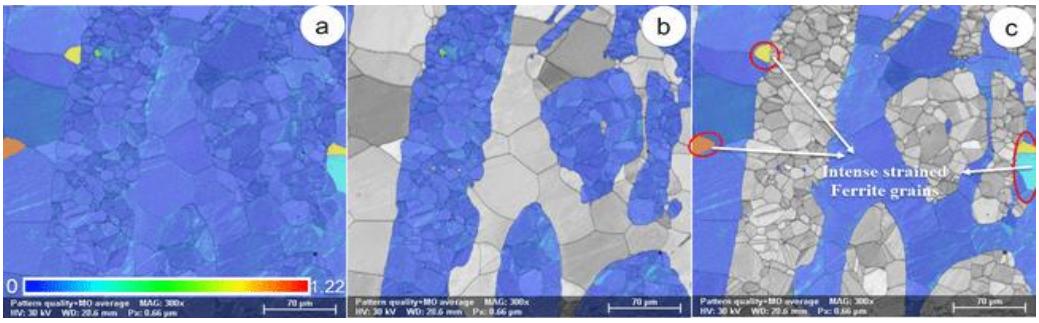

Fig.8. a) MO of the sample heat treated at 1050°C with maximum value of misorientation angle, b) MO of austenite phase, c) MO of ferrite phase

The temperature of solution treatment was increased to 1100 °C with duration 20 minutes, and microstructure was analyzed. It can be observed that the sigma phase was disappeared in microstructure because of solution treatment temperature was more than 1000°C. As well as there is an increasing of austenite volume fraction grains to (57.3%) with elongated grains, decreasing ferrite phase volume fraction to (42.7%) with bigger elongated grains and secondary ferrite phase generated in the alloy with small amount, in comparison with hot rolled sample, as shown in Fig. 9.

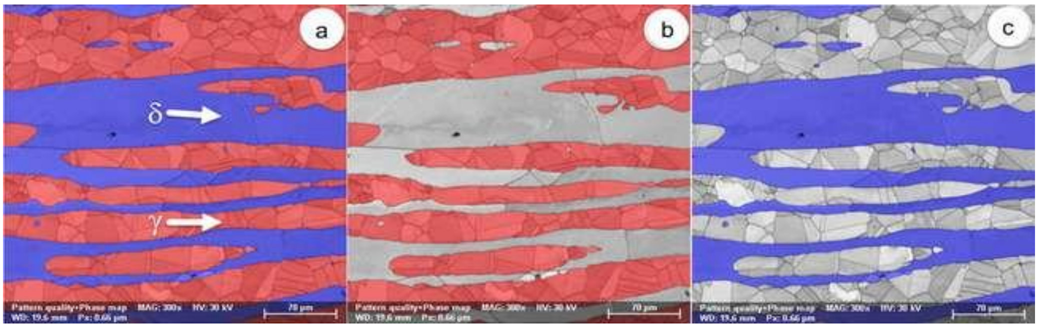

Fig.9. SEM microstructure analysis of sample heat treated at 1100°C; a) distribution of the phases, b) austenite phase deformation, c) ferrite phase deformation

Fig. 10 shows the MO average for heat treated sample at 1100°C, the misorientation angle was decreased to 1.4º in comparison with hot rolled sample, and bigger than the MO of heat treated sample at 1000°C as well as heat treated sample at 1050 °C in the same duration. It was observed that the grains in ferrite phase almost in the limit of modal orientation average and some grains in the austenite phase have high strain level as shown in Fig. 9b.

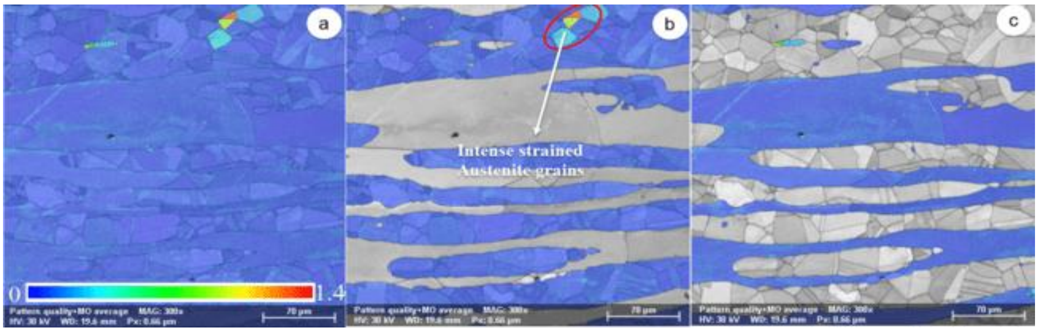

Fig. 10.a) MO with value of misorientation angle, b) MO of austenite phase, c) MO of ferrite phase.

3.2. Mechanical test (Tensile strength test)

The resulted morphologies in the present work are distinguished by different features as discussed above. Therefore, the mechanical properties of super duplex stainless steel (SDSS) F55 alloy are a consequence of its microstructure. All the samples were subjected to mechanical investigation through the tensile strength test. For each sample, the tensile test parameters: Ultimate Tensile Strength (UTS), Yield Strength (YS), and Fracture Elongation (εf) were given in Table 2. The graphs (Stress-Strain) for all samples as shown in Figs. 11.

Table 2

Mechanical properties

	Structural state				
	As-received (**AR**)	Hot-deformed: 1000°C (**HR**)	Solution treated: 1000°C-20min-WQ (**ST1**)	Solution treated: 1050°C-20min-WQ (**ST2**)	Solution treated: 1100°C-20min-WQ (**ST3**)
UTS (MPa)	733,44	768,16	771,12	742,02	744,95
YS at 0.2% (MPa)	475,47	505,29	515,78	425,52	462,28
εf (%)	55,5	34,2	46,8	46,8	54,9

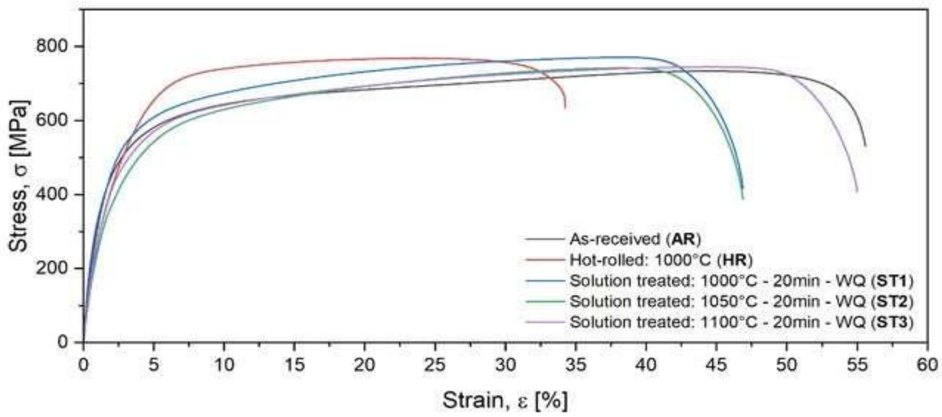

Fig.11. Tensile Stress-Strain curve of samples

Due to the hot rolling process, the UTS and YS at 0.2 % are increased, while εf (%) decreased because of generating sigma phase in microstructure of the material with volume fraction around 3.55 as well as increasing the dislocation density in the microstructure. However, the austenite phase is increased to 59.7 %. By applying the solution treatment at 1000°C for 20 minutes, the strength properties and the ductility are increased in the solution-treated samples compared to the hot-rolled sample due to recrystallizing the microstructure and decreasing volume fraction of sigma phase to (3.50%), As well as increasing volume fraction of austenite phase to (62.3%). With raising the temperature of solution treatment to 1050°C, the strength properties are decreased while the ductility value remain constant in the solution-treated samples compared to the sample which is heat treated at 1000°C due to absence of sigma phase from the microstructure and decreasing volume fraction of the austenite phase to (51.5%). At 1100°C temperature of solution treatment, the strength properties are increased also the ductility is increased in comparison with sample which was heat treated at 1050°C with continuing absence of sigma phase and austenite volume fraction is increased to (57.3).

4. Conclusion

The effects of solution heat treatment temperature upon evolution of microstructure and mechanical properties for a Super Duplex Stainless Steel UNS 32760 F55 alloy were investigated. Obtained microstructure after hot deformation and solution treatment at 1000°C showed a mixture of ferrite, austenite, and sigma phases. In the case of solution treatments at temperatures above 1050°C the deleterious sigma phase is no longer observed. Investigations on mechanical properties showed that increasing solution temperatures increase elongation to fracture (εf) from 34.2% of hot rolled sample to 54.9% of sample which was heat treated at 1100°C, while the resistance properties are decreased such as ultimate tensile strength (UTS) decreased from 768.16 MPa of hot rolled sample to 744.95 of sample which was heat treated at 1100°C sample, also yield strength at 0.2% (YS) decreased from 505.29 MPa of hot rolled sample to 462.28 MPa of sample which was heat treated at 1100°C, all due to the presence/absence of deleterious sigma phase and volume fraction of austenite and ferrite phases . Therefore, the best temperature of solution treatment to get an alloy almost like as received alloy is 1100°C.

R E F E R E N C E S

[1]. *NILSSON, J.O*. Super duplex stainless steels. J. Material Science Technology. **1992**, 8, 685-700.

[2]. *J.R. DAVIS*, ASM Specialty Handbook-Stainless Steels, ASM International, Materials Park, OH, 1996, ISBN: 0- 87170-503-6

[3]. *TEHOVNIK, F.; ARZENSEK, B.; ARH, B.; SKOBIR, D.; PIRNAR, B.; ZUZEK, B.* Microstructure evolution in SAF 2507 super duplex stainless steel. Material technology. **2011**, 45, 339–345.

[4]. *WANG, X.; CHEN, W.* Influence of cerium on hot workability of 00Cr25Ni7Mo4N super duplex stainless steel. J. Rare Earths **2010**, 28, 295–300.

[5]. *BASTOS, I.N.; TAVARES, S.S.M.*; DALARD, F.; NOGUEIRA, R.P. Effect of microstructure on corrosion behavior of super duplex stainless steel at critical environment conditions. Scr. Mater. **2007**, 57, 913–916.

[6]. *YOUSEFIEH, M.; SHAMANIAN, M.; SAATCHI,* A. Optimization of the pulsed current gas tungsten arc welding (PCGTAW) parameters for corrosion resistance of super duplex stainless steel (UNS S32760) welds using the Taguchi method. J. Alloy. Compd. **2011**, 509, 782–788.

[7]. *ARMAS, A.F.; HERENU, S.; ALVAREZ-ARMAS, I.; DEGALLAIX, S.; CONDO, A.; LOVEY, F.* The influence of temperature on the cyclic behavior of aged and un-aged super duplex stainless steels. Material Science Engineering, a **2008**, 491, 434–439.

[8]. *ARGANDONA, G.; BIEZMA, M.V.; BERRUETA, J.M.; BERLANGA, C.; RUIZ, A.* Detection of Secondary Phases in UNS S32760 Super duplex Stainless Steel by Destructive and Non-destructive Techniques. J. Material Engineering Perform. **2016**, 25, 5269 5279.
[9]. *GUO, Y.; HU, J.; LI, J.; JIANG, L.; LIU, T.; WU, Y*. Effect of Annealing Temperature on the Mechanical and Corrosion Behavior of a Newly Developed Novel Lean Duplex Stainless Steel. Materials **2014**, 7, 6604–6619.
[10]. *MA, M.; DING, H.; TANG, Z.; ZHAO, J.; JIANG, Z.; FAN, G*. Effects of Temperature and Strain Rate on Flow Behavior and Microstructural Evolution of Super Duplex Stainless Steel under Hot Deformation. J. Iron Steel Res. Int. **2016**, 23, 244 252.
[11]. *DENG, B.; JIANG, Y.M.; GAO, J.; LI, J*. Effect of annealing treatment on microstructure evolution and the associated corrosion behavior of a super-duplex stainless steel. J. Alloy. Compd. **2010**, 493, 461–464.
[12]. *TAN, H.; JIANG, Y.; DENG, B.; SUN, T.; XU, J.; LI, J*. Effect of annealing temperature on the pitting corrosion resistance of super duplex stainless steel UNS S32750.Journal Material. **2009**, 60, 1049–1054.
[13]. *WEBER, L.; UGGOWITZER, P.J.* "Partitioning of chromium and molybdenum in super duplex stainless steels with respect to nitrogen and nickel content". Material Science engineering. A **1998**, 242, 222–229.
[14]. *SOUTHWICK, P.D.; HONEYCOMBE, R.W.K.* "Precipitation of M23C6 at austenite/ferrite interfaces in duplex stainless steel". Metal science. **1982**, 16, 475–482.
[15]. *ALTURAIHI SALEH, et al*. Microstructural changes occurred during hot deformation of SDSS f55 (super-duplex stainless steel) alloy. U.P.B. Sci. Bull., Series B, Vol. 81, Iss. 1, 2019