Effect of Thermomechanical processing on Microstructure and Mechanical Properties of a Duplex Stainless Steel

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ABSTRACT

Duplex stainless steels (DSS_S) have a microstructure composed of ferrite and austenite phases that gives them a very good combination of mechanical and corrosion properties. These steels are desirable for many applications in chemical and petrochemical industries. In the present study, a type of stainless steel was cast, solution annealed at 1200 °C for 60 minutes, and then quenched in water. The initial microstructure was composed of about 51.8% ferrite and 48.1% austenite. The steel was then cold rolled about 90% and subsequent annealing at 800,850 and 900 °C for different times was performed. Results showed that tensile strength of steel hassignificantly improved. Austenite grain size of the cast steel was refined through static recrystallization of the austenite occurred during post cold deformation annealing. Results also showed that the yield strength of the as-cast steel increased from 552 to 1800 and its tensile strength increased from 672 to 2350 MPa as a result of large cold deformation. With increasing the annealing time, sigma phase increases, which leads to the decrease of elongation. Thus, the sigma phase can be expected to embitter the alloy.

1. Introduction

$DSS_s$are Fe-Cr-Ni alloys, containing 17-30% Cr and 3-31% Ni. These steels have two phases in microstructure, ferrite and austenite, where both phases are present more than 30%. The duplex microstructure is obtained by controlled alloying [1]. DSS has higher strength than the austenite grades as well as higher toughness than the ferrite ones. So, these steels have more applications in places that need high strength and toughness. The important application of these steels is in strategic industries. In addition to ferrite and austenite, a variety of secondary phases such as sigma, chi, laves, and so on may form in the temperature range from 300 to $1000^{\circ}C$ [2]. The amount of precipitations depends on temperature, time and chemical composition of steel[2-3]. Among the phases, sigma is the most important due to the fact that it has fast formation kinetics in a wide range of temperatures and it is a hard phase [2-3]. These steels are important in industries and further investigation on the relation between microstructure and mechanical and metallurgical properties is still needed. Depending on the applied thermomechanical

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Table 1. Chemical composition of the investigated steel (wt %)

Cr	Ni	Mo	Mn	C	Cu	P	Si	Fe
21	6.4	2.8	0.67	0.067	0.052	0.035	0.25	Bal.

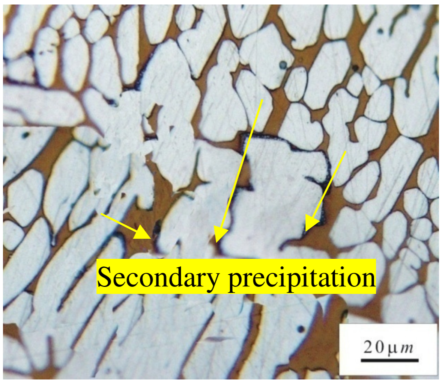

Fig. 1. Microstructure of the as-cast DSS

processing and annealing temperature and time, DSSs have different mechanical properties. On the other hand, appropriate treatments are necessary for optimization of mechanical properties. In the present study, duplex stainless steel was cast and then the material underwent thermomechanical processing. The aim of the current investigation is to study the effect of this process parameters on the microstructure and mechanical properties.

2. Experimental

Chemical composition of the investigated steel is listed in Table 1. This steel is the as-cast DSS and employed for our experiments. Chemical analysis tests of investigated steel were performed in Jahad Tahghighat Sahand Metallurgy Lab.

This steel was solution annealed at 1200$^{\circ}C$ for 60 min and then quenched in water. Finally, the specimen was cold rolled to about 90%. For investigation on the microstructure and tensile properties, specimens were prepared from cold rolled steel and then they were annealed at 800,850 and 900$^{\circ}C$ for 5 and 20 min. The specimens were subjected to standard grinding and polishing. Then, they were etched with Beraha's echant (1g $K_2S_2O_5$ + 20ml HCl + 100ml H_2O) and were studied by Olympus PMG optical microscope. Vickers hardness test was performed with 30kgf by ESEWAY plant model DVRB-M. The tensile test specimen was prepared using Jis Z2201 standard (×0.4 all of dimensions). Tensile tests were carried out using tensile machine Zwick/Rael Z010 with crosshead velocity of 0.1 mm/min.

3. Results and Discussion

The microstructure of the as-cast DSS is shown in Fig. 1. It can be clearly seen that the bright phase is austenite and the gray phase is ferrite [4]. The volume fractions of ferrite (α) and austenite (γ) are about 35% and 65%, respectively. These results were calculated by Clemex Image Analysis software.

Secondary precipitations are visible in the boundaries between austenite and ferrite in the as-cast steel. The removal of secondary precipitation from the cast materials is usually performed through a solution annealing heat treatment. The solution annealing heat treatment reaches a high enough temperature to completely dissolve secondary precipitation and the steel is then rapidly cooled to ensure that secondary precipitation does not reform [5]. Of course, the aim of solution annealing is notonly to removesecondary precipitation but also to balance ferrite-austenite ratio. The ferrite content in the microstructure depends on the solution annealing temperature. As the temperature increases, the ferrite concentration increases [6].

Fig. 2 shows the as-cast steel microstructure after solution annealing and water quenching. The volume fraction of austenite and ferrite are 51.8 and 48.1, respectively. So, austenite is transformed to ferrite with increasing the temperature of the as-cast DSS up to1000$^{\circ}C$.

The microstructure produced after cold rolling is shown in Fig 3. In this figure, the lighter phase is austenite and the darker one is ferrite. It can be seen that during rolling both phases are aligned and hard worked.

X-ray diffraction spectra of the cold rolled DSS (Fig. 4) shows peaks of austenite and ferrite with no extra peaks corresponding to other phases, indicating that there might be no or only slight amounts of other precipitation

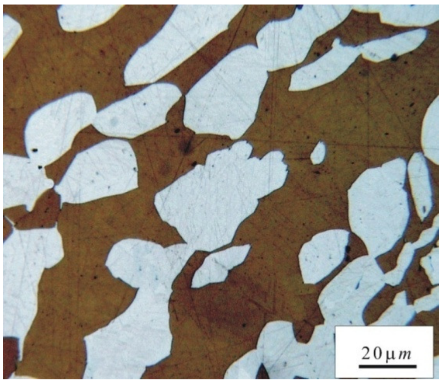

Fig. 2. Microstructure of the as-cast DSS after solution treatment at 1200$^{\circ}C$ for 60 min and water quenching

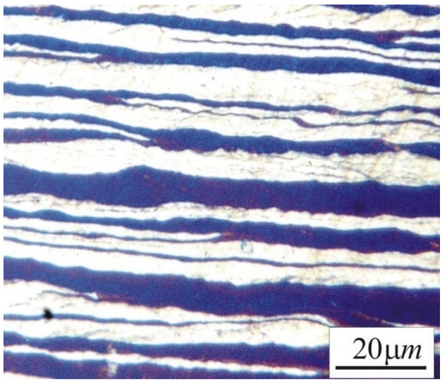

Fig. 3. Microstructure of the rolled DSS (TD direction)

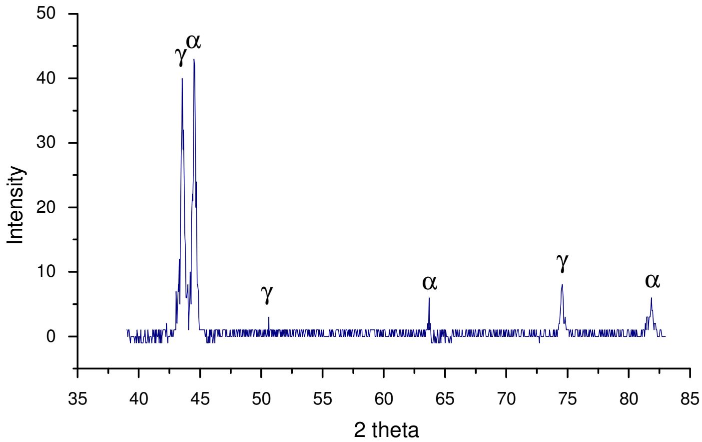

Fig. 4. X-ray diffraction of the cold rolled specimen

present. Results of microstructure and XRD analyses of the cold rolled sample shows the presence of the two phases austenite and ferrite with no other phase being detected. The microstructures of annealed DSS at different temperatures for 5 and 20 min are shown in Figs. 5 and 6.

As it is shown in these figures, decomposition of the ferrite phase into other phases is taking place at all three temperatures. Secondary formed phases after annealing were distinguished by X-ray diffraction (Fig. 7). The X-Ray spectra of the specimens annealed in the temperature range from 800 to 900$^{\circ}C$ are similar and showed the presence of three phases: ferrite, austenite and sigma (σ). It can be seen that the peaks of sigma were detected at three temperatures. The mechanism of σ phase precipitation is a eutectoid reaction $\alpha \rightarrow \sigma + \gamma_2$ at temperature ranging from 600 to 1000$^{\circ}C$. Relationship between the intensity of the peaks corresponds to ferrite and sigma phase observed in these diagrams.

An increase in the number and intensity of the sigma peak is accompanied by a decrease of the ferrite peak height. Ferrite is transformed into the sigma and secondary austenite phase according to the eutectoid reaction $\alpha \rightarrow \sigma + \gamma_2$. The high content of chromium and molybdenum in the ferrite phase presumably causes the formation of the σ phase at the expense of ferrite [7] because σ phase is rich in Cr and Mo.

Fig.7 also shows that the number and intensity of the sigma peaks at 850$^{\circ}C$ are more than the other two temperatures, so that transformation in this temperature occured with most intensity. The fastest precipitation rate for

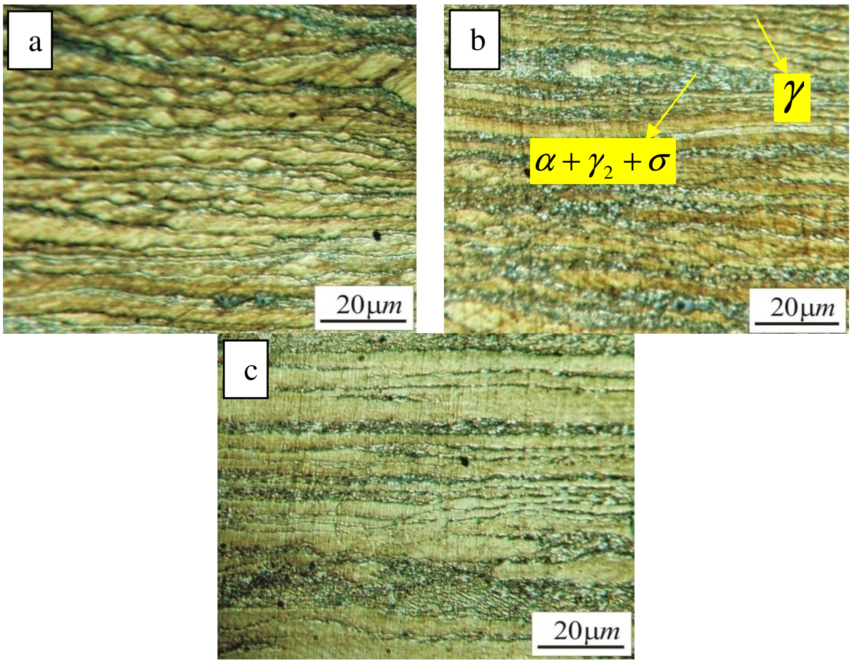

Fig. 5. Microstructure of the cold rolled DSS with annealing at temperature a) 800, b) 850, and c) 900 $^\circ C$ for 5 min

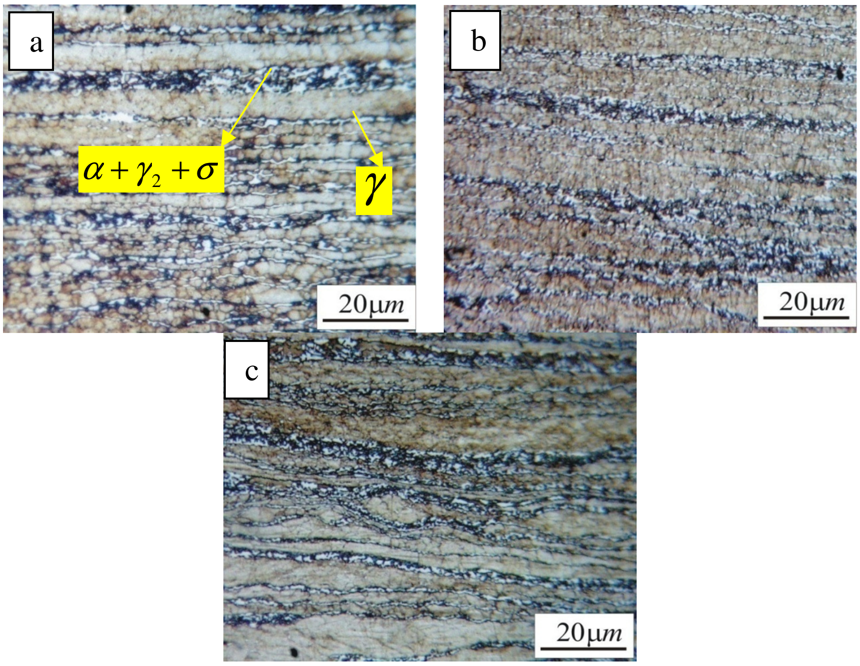

Fig. 6. Microstructure of cold rolled DSS with annealing at temperature a) 800, b) 850, and c) 900 $^\circ C$ for 20 min

σ phase can be seen at 850 $^\circ C$, i.e., the nose of Time-Temperature-Transformation curve for this steel is at 850 $^\circ C$. The ferrite phase is transformed because of the fact that the diffusion rate of the alloying elements in the ferrite is 100 times faster than the corresponding values in the austenite [8]. This is a consequence of the less compact lattice in the bcc crystal structure. Annealing in per three temperatures for 5 min is lower than 20 min subjected under transformation and sigma phase formed very low, because the ferrite transformation is time depended [9-10].

X-ray diffraction of the samples annealed at

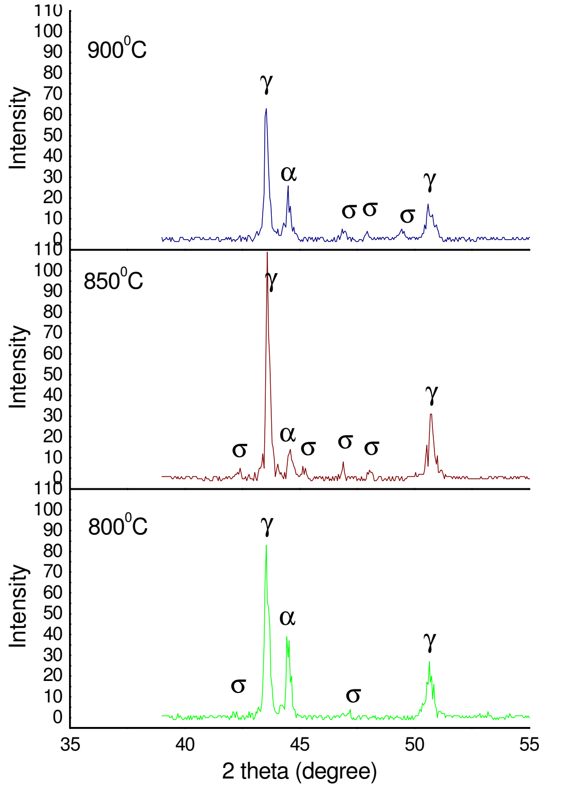

Fig. 7. X-ray diffraction of the samples annealed at 800,850 and 900 $^{\circ}C$ for 20 min

800 $^{\circ}C$ for 5 min (Fig. 8) showed lower numbers and intensities of the sigma phase peaks. The volume fraction of the sigma phase increases after cold rolling with increasing the annealing time.

As can be seen in Fig. 6, in the samples annealed for 20 min static recrystallization has taken place and the grains have been smaller, but in the annealing for 5 min there is not enough time for such an occurrence. Studies show that phase transformations are necessary to obtain finer structure during thermomechanical processing in the two-phase alloys [11]. In the two-phase alloys recrystallization the phases are often independent from each other. Austenite phase recrystallized because the austenite grains saved much energy and were qualified for nucleation and recrystalization [11]. As can be seen in fig.6, the austenite grains begin to grow at 900 $^{\circ}C$ because the structure was not thermodynamically stable after the completion of recrystallization. Therefore, if conditions improve, recrystallized grains will grow. The driving force for this process is decreasing energy saved in the grain boundaries in the material [11]. Austenite grains in the sample annealed at850 $^{\circ}C$ for 20 min are smaller than those at800 $^{\circ}C$. The austenite phase in the casting state was related to islands with 25 micrometer in size. The austenite sizes of equiaxed grains with annealing for 20 min at 800, 850 and 900 $^{\circ}C$ were 4, 2.53, and 5.6 micrometer, respectively. The grain size in the casting is coarser than in the mechanically deformed structure.

Hardness and tensile tests were performedin order to evaluate mechanical properties related to microstructural changes after annealing. Stress-strain curves of the cold rolled sample and the cast steel are shown in Fig. 9.

The ultimate tensile strength, yield strength and the elongation are calculated before and after the rolling process. Before the rolling process, ultimate tensile strength, yield strength and elongation are 672,552 MPa, and 42%, and after rolling are 2350, 1800 MPa, and 24%, respectively. By comparing these results,it can be concluded that cold work increased the sources of dislocation and, as a consequence,a high density of dislocations was generated and their movement resulted in plastic deformation. High dislocation density in material increases strength and yield stress and decreases toughness [11]. Fig. 10 shows engineering stress-strain curve of the rolled sample annealed under different conditions.

The ultimate tensile strength, yield strength and the elongation are 1933, 1500 and 35%, respectively, for sample with annealing for 5 min at 800 $^{\circ}C$. Annealing resulted in the release of some of dislocations so the strength decreased and elongation increased when no annealing was used.

The amounts of ultimate tensile strength, yield strength and elongation of samples annealed for 20 min at 800,850 and 900 $^{\circ}C$ were calculated and shown in Fig. 11.

Ultimate tensile strength and yield strength of the specimens increased with increasing the annealing temperature from 800 $^{\circ}C$ to 850 $^{\circ}C$.

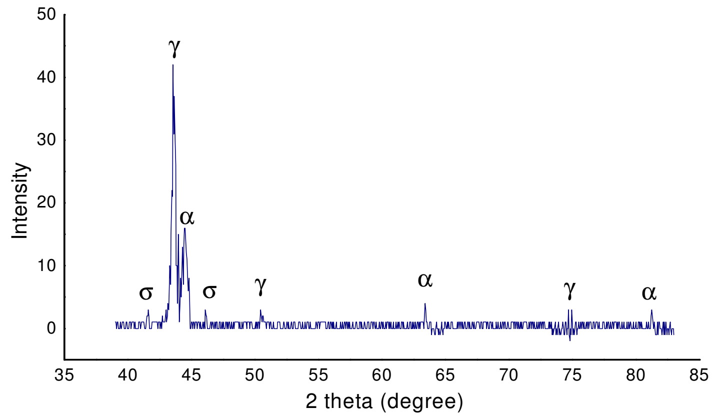

Fig. 8. X-ray diffraction of sample annealed at $800^{\circ}C$ for 5 min

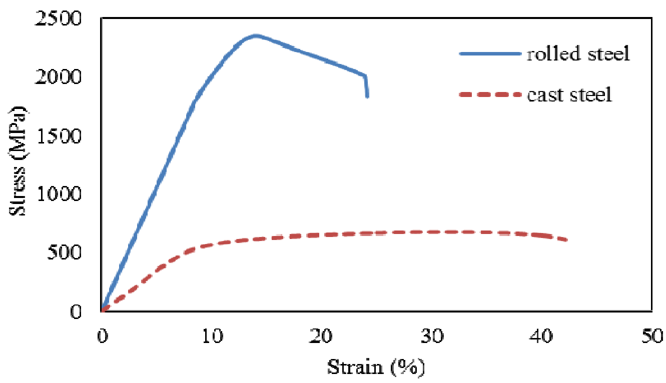

Fig. 9. Engineering stress-strain curve of the cold rolled sample (without annealing) and the cast steel

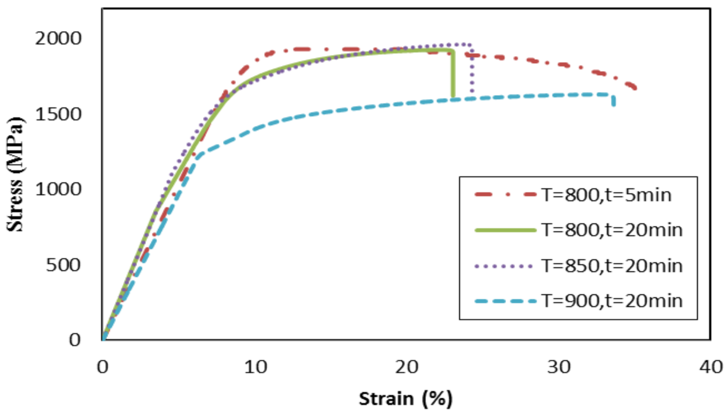

Fig. 10. Engineering stress-strain curve of the cold rolled and annealed samples

Also, ultimate tensile strength and yield strength decreased with increasing the temperature to $900^{\circ}C$. These changes are due to microstructure transformation. As discussed above, Austenite grains are smaller at $850^{\circ}C$. The presence of fine-grained microstructure generally proves the mechanical properties of DSS. Grain refinement is usually obtained in the as-cast steel by means of an adequate thermomechanical process. Thermomechanical processing, which combines deformation and heat treatment, is very effective for microstructure control and hence for the

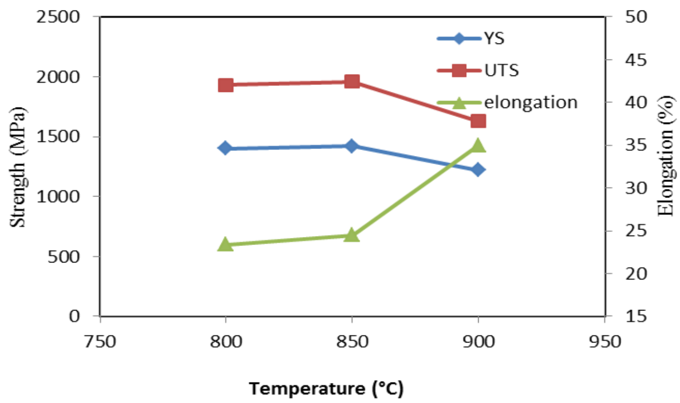

Fig. 11. Ultimate tensile strength (UTS) and yield strength (YS) and elongation of the samples annealed for 20 min at 800,850 and 900 $^\circ C$

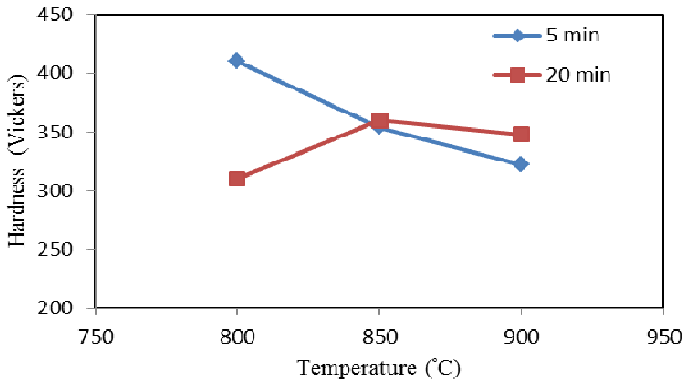

Fig. 12. Hardness of the samples annealed for 5 and 20 min at 800, 850, and 900 $^\circ C$ after rolling

improvement of mechanical properties of metallic materials. Also, it is known that the yield and ultimate tensile strengths increase with decreasing the grain size according to the Hall-Petch relation. Austenite grains grow with increasing temperature from 850 $^\circ C$ to 900 $^\circ C$ so the strength decreased. Also, the sigma phase at 900 $^\circ C$ is lower than thatat 850 $^\circ C$.

The effect of annealing temperature on elongation showed that decreasing of the sigma phase resultedin the increase of elongation. Because the sigma phase is brittle and hard, it affects toughness. Such results demonstrate that the sigma phase can be expected to embitter the alloy. Values of samples hardness with rolling and annealing for 5 and 20 min at 800,850 and 900 $^\circ C$ are shown in Fig 12. Hardness of the rolled sample without annealing was 450 Vickers. It increases in comparison with the sample without rolling (250 Vickers). This is due to severe cold work during cold rolling.

Hardness is strongly related to the percentage of the sigma phase formed [12-14], but this is not the only factor which must be taken into account. Annealing treatments also induce changes on the microstructure, internal stresses, dislocation and crystallographic textures which could influence the hardness of DSS. It can be seen that hardness in the samples annealed for 5 min decreased with increasing the annealing temperature due to decreasing dislocation density, but in samples annealed for 20 min the hardness at 850 $^\circ C$ is more than other temperatures due to the fact that the sigma phase at this temperature is more than at other temperatures. The sigma phase increased hardness due to brittle nature and high hardness. Fig. 13 shows scanning electron micrograph (SEM) of fracture surfaces of the (after tensile test) rolled samples without

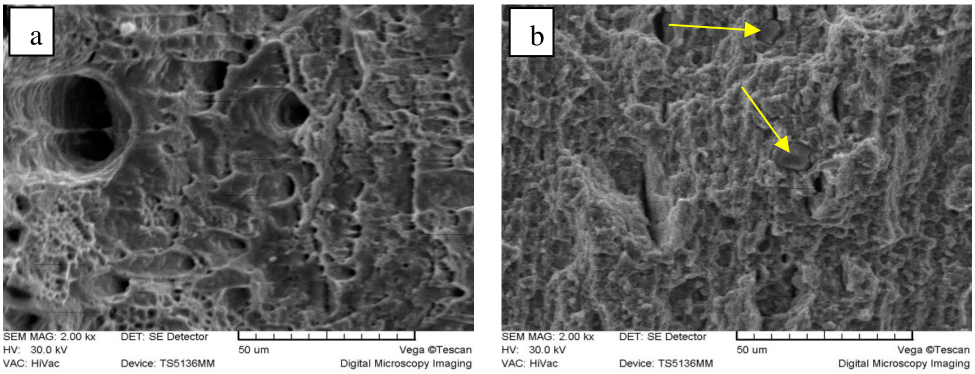

Fig. 13. Fracture surfaces of rolled samples: a) without anneal, b) annealed for 20 min at 800 $^{\circ}C$

anneal and annealed for 20 min at 850 $^{\circ}C$.

In Fig.13a, dimples are distributed over the surface,which is the characteristic of ductile fracture. Big dimples were observed in the fracture surface. They were formed from voids nucleation and growth. Some phases are observed that are probably sigma phase and are responsible for brittle fracture in this sample, as shown by arrows in Fig. 13b.

4. Conclusions

In this study, Fe-21 Cr-6.4 Ni-2.8 Mo DSS was cast, cold rolled after solution treatment and subsequently annealed at 800,850 and 900 $^{\circ}C$ for 5 and 20 min. Theobtainedresultsare summarized as follows.

1. Ultimate tensile strength and yield strength of the studied steel increased with cold rolling.
2. Metallography studies and X-ray diffraction results show the presence of the sigma phase during annealing at 800,850 and 900 $^{\circ}C$. Sigma phase at 850 $^{\circ}C$ is more than at the other two temperatures.
3. Static recrystallization of the material occurs after annealing for 20 min, so the austenite phase becomes micrograin size austenite. i.e., the grain size in the casting is coarser than that in the mechanically deformed structure.
4. In the casting state, the austenite phase was islands but Austenite phase with annealing for 20 min at 800,850 and 900 $^{\circ}C$ wasequiaxed.

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