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Microstructural Evolution and Mechanical Properties of Type 304 L Stainless Steel Processed in Semi-Solid State

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Abstract The paper discusses the effect of semi-solid processing on the microstructural evolution and mechanical property of 304L stainless steel. For the study, steel specimens were partially melted and cooled to room temperatures in different cooling medium. The effect of temperature, time and cooling medium on microstructural evolution was studied by using optical microscopy. It was found that melting begins with the nucleation of liquid phase at the triple junctions and grain boundaries followed by propagation of the liquid phase along grain boundaries. The mechanical behavior of the semi-solid processed material was compared with that of the conventionally processed material with regard to their tensile properties and hardness. The semi-solid processed material shows better ductility and reduced YS and UTS than the conventionally processed counterpart. The correlation of the tensile properties and evolved liquid content shows that the UTS of the material decreases, while the YS increases with liquid fraction. The study also shows that there is a significant effect of cooling medium on the microstructural evolution and hence on the mechanical properties.

Keywords 304L Stainless Steel, Semi-Solid Processing, Microstructure, Tensile Properties, Hardness

1. Introduction

AISI type 304L austenitic stainless steel (304L SS) is widely used in power, chemical, petrochemical[1] and nuclear industries[2]. Conventionally, stainless steels are fabricated by casting and forging processes. However, continuous efforts are being made to find alternatives manufacturing routes to accomplish many objectives such as reducing energy consumption, enhancing workability, producing near-net shape products, and minimizing intermediate process steps[3]. Semi-solid processing is one such promising alternative to conventional manufacturing techniques[4], which not only fulfills the above objectives but also combines the benefits of both conventional casting and forging processes. This technology has already been adopted in an industrial scale for manufacturing of various non-ferrous alloys[4], such as aluminum[5] and magnesium alloys[6, 7]. The suitability of this technology has also been investigated for producing some components in laboratory scale from the ferrous alloys, such as X210Cr[3, 8] and bearing steels[9]. Therefore, adoption of this technology for other grades of steels has drawn worldwide attention. The major constraints in semi-solid processing of steels arise from high melting point of these alloys, narrow semi-solid temperature range, and phase transformation during melting and solidification[10]. In addition to the processing-related issues, assessing the suitability for semi-solid processing of steels also requires evaluation of post-processing mechanical properties. Therefore, the present work focuses on the microstructural evolution in 304L SS after cooling from the semi-solid state and on the resultant mechanical properties. A comparative study of the microstructure and mechanical properties of semi-solid and conventionally processed 304L SS is presented and discussed.

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2. Experimental

For the present study, commercially available solution annealed 304L SS plates, with chemical composition Fe-18.52Cr-7.85Ni-0.016C-1.59Mn-0.29Si-0.003S-0.016P, have been used.

The microstructure of the steel in as-received condition is shown in Figure1. Small bars of size 15mm × 15mm × 120mm were cut from the plates and used to carry out the experiments in the semi-solid state. The bars were partially melted in a muffle furnace and then allowed to cool to room temperature inside the furnace. To investigate the effect of higher heating and cooling rates on the microstructural features of the material, a few cylindrical specimens of size of 10 mm diameter and 15 mm height were heated in a Gleeble thermo-mechanical simulator to the semi-solid

temperature, held at the targeted temperature for 30s, and then quenched in water. The experimental details are given in Table.1 After cooling the bars and cylinders to room temperature, a small sample of each specimen was taken for microstructural investigation. The diamond polished metallographic samples were etched in 10% oxalic acid solution at 2V, and microstructures were observed using an optical microscope.

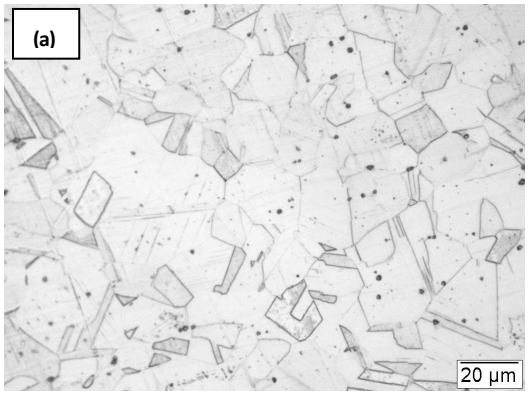

Figure 1. Microstructure of solution annealed SS 304LTable 1: Parameters used for heat treatment of SS 304L

Table 1. Parameters used for heat treatment of SS 304L

Sp. No.	Heating Rate (°C/sec)	Temper -ature (°C)	Soaking Time (mins)	Cooling Condition
S1	300	1400	5	Furnace Cooled
S2	300	1400	15	Furnace Cooled
S3	300	1400	25	Furnace Cooled
S4	300	1410	5	Furnace Cooled
S5	300	1420	5	Furnace Cooled
G1	5	1400	½	Water quenched
G2	5	1410	½	Water quenched
G3	5	1420	½	Water quenched

The mechanical properties of the material processed in semi-solid state were evaluated by carrying out tensile tests at strain rates 0.01 s^{-1} at room temperature using a 100kN tensile testing machine. Standard (as per ASTM standard E8M) specimens fabricated from furnace-cooled bars were used for the tensile tests. Vicker's hardness of the furnace-cooled bars as well as the water-quenched specimens was measured in hardness testing machine under a load of 10 kg.

3. Results and Discussions

3.1. Microstructural Evolution

Microstructural evolution of any material depends on its chemical composition[11] and various parameters defined by the processing route such as heating rate, temperature of processing[12], cooling rate[13], mechanical loading[12], and so on. In any investigation on the effect of the processing route or the parameters involved in the process on the microstructure, it is always necessary to understand the effect of composition, as it defines the various phases that form within the processing temperature regime. In semi-solid processing, the material is heated to a temperature between the solidus and liquidus temperatures and then cooled to room temperature. Hence, it is of paramount interest to know the possible phases that can form between room temperature and the liquidus temperature. It is well known that the 304L SS retains its austenite phase at room temperature and undergoes phase changes at high temperatures, depending on the Cr-equivalent (Cr_{eq}) to Ni-equivalent (Ni_{eq}) ratio of the steel[14]. In order to calculate the Cr_{eq} and Ni_{eq} for different grades of 304 SS, the following equations are used[14]:

$$Ni_{eq} = Ni + 30 \times C + 30 \times N + 0.5 \times Mn \quad (1)$$

$$Cr_{eq} = Cr + Mo + 1.5 \times Si + 0.5 \times Nb \quad (2)$$

where Ni, C, N, Mn, Cr, Mo, Si and Nb are the wt% of the elements present in the steel. Using the above equations, it was found that the Cr_{eq}/Ni_{eq} ratio for the present grade of 304L SS is 2.02. This indicates that the steel under investigation follows a primary ferritic mode of solidification[14]. Upon heating, the austenite (γ) completely transforms to delta-ferrite (δ) and further heating causes melting. The transformation follows the reverse order during cooling. At the solidus temperature, the liquid phase is nucleated at triple junctions and grain boundaries depending on the solid-liquid interface energy (γ_{SL}) and the grain boundary energy (γ_{GB})[15]. With further increase in temperature, the liquid phase propagates along the grain boundaries and forms a continuous network along the grain edges. The growth of liquid phase along the grain edges causes a continuous decrease in size of the grains which gradually approach a critical size. Once the grains achieve the critical size, rapid melting occurs and the grains disappear in the molten metal pool[16]. However, in this study, the material has not been allowed to undergo complete melting. The material has only been allowed to melt partially, after which it was cooled to room temperature. The microstructures of samples that were furnace-cooled after partial melting (Figure 2) indicate the solidified fraction of the liquid around the grains.

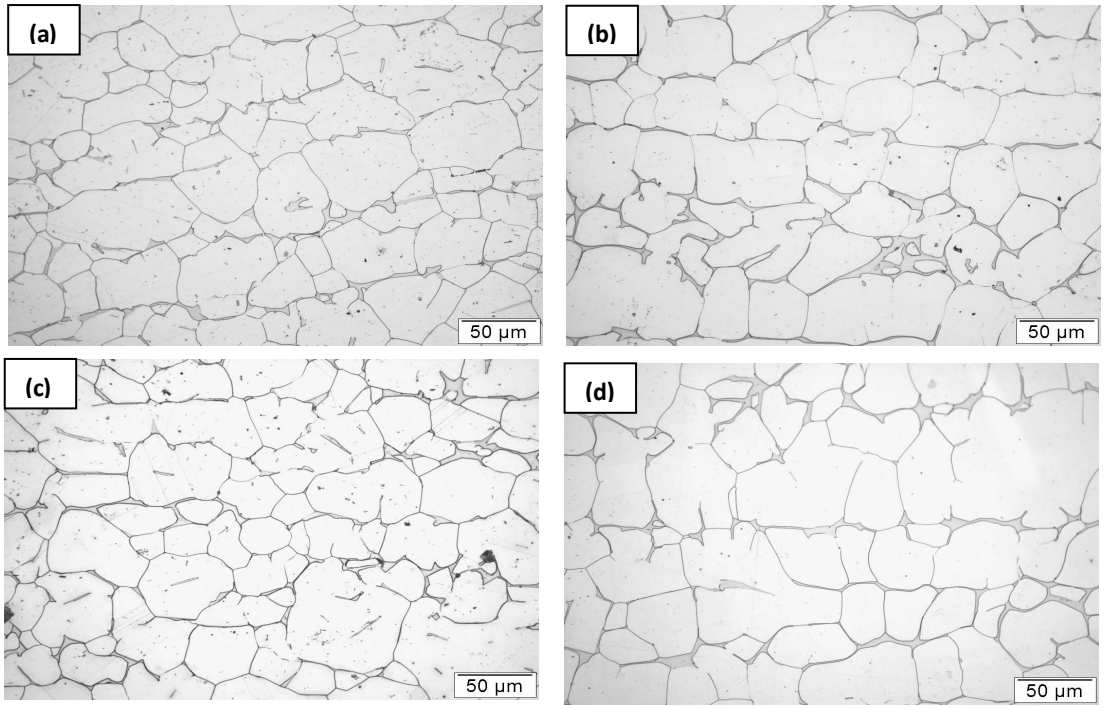

Figure 2. Microstructural evolution in 304L due to partially melting at (a) 1400°C/5 mins (b)1410°C/ 5min (c) 1420°C /5min (d) 1400°C/ 25 min followed by furnace cooling

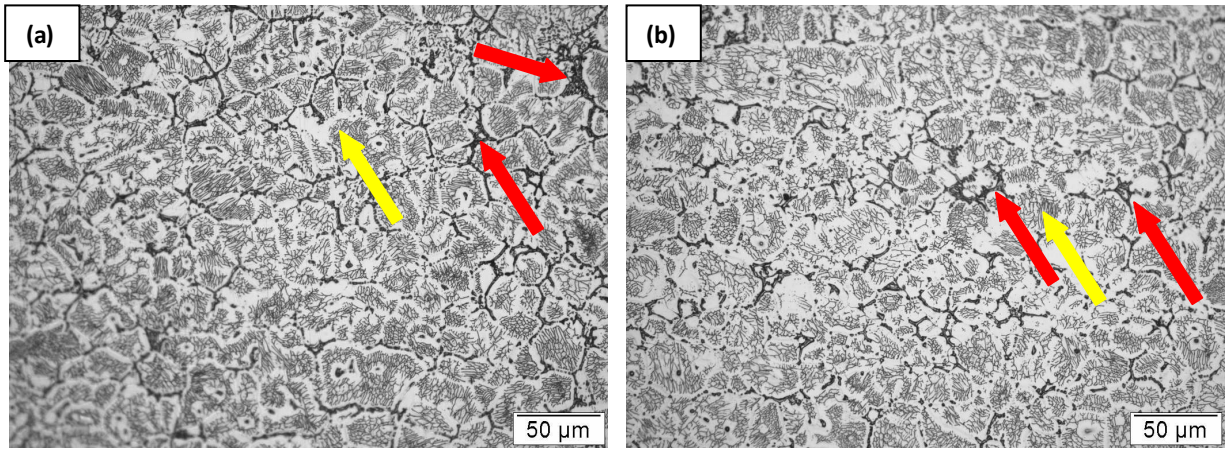

Figure 3. Microstructural evolution in 304L due to partially melting at (a) 1410°C (b)1420°C followed by water quenching

It could be observed that these samples contain bigger grains that vary from ~25 to 60µm, whereas the average grain size of the sample was 17µm before processing. This shows that grain growth occurs in the material before initiation of melting. The nucleation of the liquid phase at the triple junctions and grain boundaries can be seen in the Figure2(a), while increase in the liquid phase and its propagation along the grain boundaries with temperature and time of holding can be observed from Figs.2(b)-2(d). From these figures, it is observed that with increase in temperature from 1400 to 1420°C, there is a significant increase in the number of liquid phase entrapments inside the solid grains. However, no such increase in sites of interior grain melting was obtained with increase in soaking time. Only growth of the nucleated liquid phase by melting of the interface boundary was observed with prolonged soaking time.

On comparing the microstructures shown in Figure2, it can also be noticed that the grain edges become smoother with increase in liquid fraction. In many metals, the appearances of facets have been witnessed either on melting or on solidification[16]. The appearance or disappearance of the facets during melting and solidification depends on the thermal environment at the solid-liquid interface[17]. Though the facet disappearance is easily noticeable in the microstructure of semi-solid processed 304L SS, it is difficult to conclude that the disappearance has occurred during melting as the micrographs have been taken after solidification of the molten fraction.

The microstructure of the water-quenched samples after partial melting is shown in Figure3, in which the liquefied regions around the grains are shown by red arrows. A visual comparison of these micrographs with those of the

furnace-cooled samples indicates that the liquid fraction is relatively low in the water-quenched samples. This could be attributed to the smaller holding time of the samples at the melting temperature. The other noticeable features in these micrographs are the small network of delta ferrite inside the grains (shown by yellow arrows). The occurrence of the fine delta-ferrite networks could be ascribed to the insufficient time for the $\delta \rightarrow \gamma$ transformation due to high cooling rate (~1500ºC/s) obtained on water quenching by spray nozzles in the Gleeble thermo-mechanical simulator. These delta-ferrite networks are not visible in the microstructures of the furnace-cooled samples, as in this case, the material does get sufficient time for $\delta \rightarrow \gamma$ transformation.

3.2. Mechanical Properties

Tension test is widely used to provide the important information on the strength of a material for the design of the product or for making structures using the material[18]. Therefore, to study the effect of the process parameters as well as the microstructure on the strength, it is required to evaluate the tensile properties of the material. However, it is always not possible to measure the tensile properties of a small volume of material produced in a laboratory scale. Therefore, hardness testing provides an alternative means of assessing their mechanical properties[18]. There are several relationships available to correlate the hardness with the yield strength and ultimate tensile strength of materials[18, 19]. In this investigation, both tensile properties and hardness have been evaluated for the samples that were furnace-cooled after partial melting, while only the hardness has been determined for the samples that were water-quenched after partial melting, due to size limitation of the samples used. The yield strength (YS), tensile strength (UTS) and the ductility (percent uniform elongation) obtained from the tensile tests performed on the as-received material (conventionally processed) and on the semi-solid processed material (with furnace- cooling) are given in Table 2. From the table, it is observed that the material processed in the semi-solid range shows better ductility than the conventionally processed (by hot rolling followed by solution annealing) material, i.e. However, these materials show reduced YS and UTS compared to the conventionally processed one. Similar comparison of the tensile properties of a cast product of 304L SS reported in literature[20] shows that the semi-solid processed material shows better UTS and ductility with a comparable YS value. The correlation between the fraction of liquid and the tensile properties are shown in Figure4. From this figure an overall decrease in UTS is observed over the entire domain of study, while the YS is found to have improved with increase in liquid content. On the other hand, an initial decrease in the ductility is observed up to a liquid fraction of 14% beyond which the ductility increases with increase in liquid fraction. Here, it is to be noted that the liquid fraction in the specimens have been calculated using image analysis technique. For calculation of the liquid fraction in a particular specimen, an average of 15 micrographs of each specimen has been used. The micrographs have been taken from various locations of the specimen at different magnifications. The errors involved in the calculation could be minimized by carrying out thermal analysis of the material in the solidus–liquidus temperature domain. Table 2 also shows hardness values of these materials. The hardness values of the furnace-cooled material are found to be lower than those of the as-received material; while these are found to be similar to the hardness value of the cast steel. The variation of hardness with the liquid fraction shows a similar trend as that of the YS. These results agree with the earlier findings on the relationship between the hardness and YS of austenitic steels[19].

Table 2. Mechanical properties of the conventional and semi-solid processed specimens

	Liquid Fraction	YS (MPa)	UTS(MPa)	Uniform Elongation (%)	VHN (10kg)
As-Recieved		275	715	58	186
S1	10±2%	205	682	75	144
S2	13±3%	220	664	70	134
S3	16±4%	235	671	68.5	131
S4	14±2%	246	652	67	157
S5	21±3%	254	651	70	143
G1		-	-	-	162
G2		-	-	-	162
G3		-	-	-	165
CF-3[20]		248	531	60	140

On comparison of the hardness values of furnace-cooled specimens with the specimens that were water quenched after partial melting, it is noticed that the water-quenched specimens are harder than the furnace-cooled ones. As the hardness of the material reflects the trend in YS, higher YS is expected in water-quenched specimens. Higher strength of the specimens could be correlated to the presence of the fine delta-ferrite networks in the microstructure. At room temperature, a higher shear stress is required to cause slip in a bcc material than in a fcc material[18]. As delta-ferrite has a bcc structure[21], the presence of which in the fcc austenite matrix increases the average shear stress required to cause plastic deformation in the material. In addition, the presence of finely distributed second phase in the matrix is known to contribute to the strengthening of the material in many ways[22], most significantly by pining the dislocations and reducing their mobility[23]. Thus, the higher resistance to plastic deformation can be attributed to the fine network of the delta-ferrite in the austenite matrix in the water-quenched specimen. The mechanical properties of these materials have not been correlated to the liquid fraction as the evolved liquid fraction in these specimens could not be calculated using the image analysis technique owing to the presence of fine delta-ferrite networks in the microstructure. In this case also, the hardness of the semi-solid processed material is found to be less than that of the as-received material; however, its harness is higher than that of the cast 304L SS.

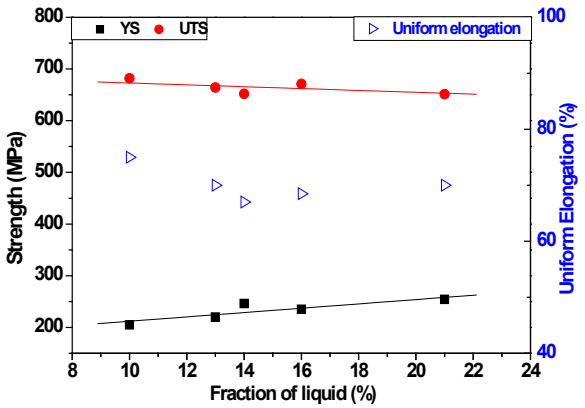

Figure 4. Correlation between the fraction of liquid and the tensile properties

4. Conclusions

The effect of semi-solid processing on the microstructural evolution and mechanical properties of a type 304L SS has been studied. Towards this end, the partially 304L SS were cooled to room temperature using different cooling medium. Tensile properties and hardness of the specimens were evaluated to compare the mechanical behaviour of the semi-solid processed material with the conventionally processed material. Also, the effect of evolved liquid content during semi-solid processing on the mechanical properties of this material has been studied. The following conclusions can be drawn from the above investigation.

1. The solidification mode of type 304L SS is primary ferrite. With increase in temperature, severe grain growth occurs in the specimen, and melting begins with the nucleation of liquid phase at the triple junctions and grain boundaries followed by propagation of the liquid phase along the grain boundaries.

2. An increase in melting temperature causes the nucleation of liquid phase at the grain interiors along with the triple junctions and grain boundaries, whereas increase in soaking time causes growth of the nucleated phase.

3. The semi-solid processed material shows better ductility and reduced YS and UTS than their conventionally processed counterpart that was solution annealed after hot rolling.

4. An overall decrease of UTS in observed over the entire domain of study; the YS is found to improve with the evolved liquid content.

5. Hardness of water quenched specimens is superior to that of furnace cooled specimens. The superior resistance to plastic deformation has been attributed to the presence of fine delta-ferrite networks in the water quenched specimens.

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