Effect of Mechanical Process on Microstructure of 316L Stainless Steel for Implant Application

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Abstrak

This research concerns the effect of rolling and bending process on microstructure of 316L stainless steel for implant application. In the present work, the as received material with initial thickness 2.0 mm was cold rolled to 70% of thickness reduction. Then, the cold rolled steels were slowly stressed using a special jig to become U-bend shape. The strain hardening was analyzed according to percent of increment with respect to longitudinal and lateral dimensions of the steel. Also, hardness measurement was used to analyze the mechanical resistance of the steel. The surface morphology of the steel was made using optical microscopic, in order to study the cold deformation effect. From the result obtained, the rolling process causes the microstructure changes by the formation of an extensive twinning boundary. Furthermore, the bending process revealed the formation of inner cracking, which is decreases the mechanical resistance of the steel.

Keywords: 316L stainless steel, rolling, bending, surface morphology, twinning boundary.

Introduction

The 316L stainless steel has been used for all major categories of implant application; orthopedic, dentistry, cardiovascular, craniofacial and otorhinology **[1]**, due to its combination of biocompatibility, corrosion resistance and acceptable mechanical strength. The successful application of the 316L grade as the metallic implants mostly depends to a proper materials selection and design, manufacturing process and the acceptance of the implant with human body environment without inverse biological effect.

Any commercial grade of 316L implants were involved a series of metal forming process during its fabrication, such as forging, drawing, rolling and bending in order to achieve the desired mechanical properties. The processes were held at the room temperature for the reason that the 316L grade can be hardened by cold work process.

Many research works have been done on the effect of cold deformation on austenitic stainless steel, specifically 316L grade. For example to study the evolution of the microstructure and texture of a AISI 316L austenitic stainless steel **[2]**; to characterize the microstructures after plastic deformation in 316L stainless steel **[3]**; to analyze the precipitation stages in a 316L austenitic stainless steel **[4]**; and to analyze the effects of plastic deformation in cold working process on the corrosion resistance, micro-hardness and mechanical properties of austenitic stainless steel.

Therefore, this research works aimed to study influence of the mechanical process on the microstructures of commercial grade of 316L stainless steel. The mechanical process consists of rolling and bending that have been carried out by cold rolled the steels up to 70% before stressed them to U-bend shape. The correlation of the microstructures to hardness properties was also investigated.

Materials and Methods

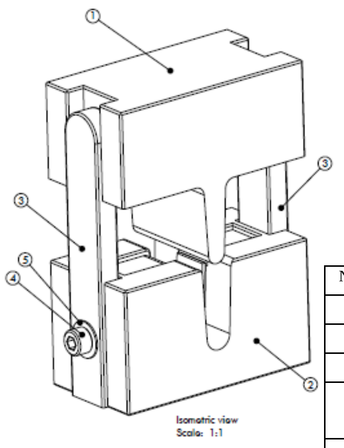
Experiments were carried out on a commercial grade of 316L stainless steel, with the chemical composition, as reported by the manufacturer, presented in **Table 1**. The as-received material, a form of sheet cutting steel, with 2 mm thickness was cut to small pieces with a dimension of 85±1 mm long and 25±1 mm width. The samples were classified according to three levels of thickness reductions, which were 1.8, 1.4 and 0.6 mm.

The rolling process was performed at room temperature with a motorized single rolling mill: model LS 120 (Italy). The machine was a bench type with the roller blade of 54 mm diameter cylinders and 120 mm long. The thickness reduction was about 0.1 mm per each pass and it took for approximately 3s. Common lubricating oil was applied to all samples before the samples passes through the roller blade for reducing the friction and wavy effects. The samples were unidirectional cold rolled to 10, 30 and 70% thickness reduction and the dimension changes of all samples after cold deformation were recorded in order to predict strain hardening effects.

Table 1
Chemical composition of the 316L stainless steel.

	Chemical Composition (%)							
316L	C	Cr	Ni	Mo	Cu	Mn	P	S
	0.02	16.6	10.4	1.08	0.28	2.03	0.03	0.002

The cold rolled samples were cut to dimensions of 80 mm long and 20 mm width, with a hole of 10 mm diameter at the both side of the sample. The samples were sandpapered and rinsed by distilled water before preparing the U-bend sample, according to ASTM G 30-97. The bending process was conducted at room temperature under a displacement control with a constant speed rate of 3 mm/min. A special jig (**Figure 1(a)**) was attached to a tensile machine, model Instron 5585 (US) with capacity of 150 kN and the stress was applied by bending the cold rolled samples to the U-bend shape. The U-bend shape was maintained so that the legs are parallel by means of bolt and nut. Also, an insulator, a recycle plastic material, was used between the sample and bolt to avoid corrosion effects. **Figure 1(b)** shows one of the actual U-bend samples used in the research.



No.	Part Name	Quantity
1	Jig Male	1
2	Jig Female	1
3	Sliding Guide	2
4	Hex Cap Screw M8 x 25	2
5	Washer M8	2

Figure 1 (a) A special bending jigs which were used to stress the cold-rolled samples; and **(b)** The actual size of the U-bend sample.

The metallographic samples were prepared by cutting the cold rolled and U-bend sample using a low speed diamond saw machine, model Labcut 1010 (UK). A small portion of the cold rolled samples, with dimensions of 2 x 2 mm, were cut using diamond saw at a speed rate of 350 to 400 rpm and a lubricating oil were continuously applied during the cutting process. The U-bend samples were cut into half of the original size by using similar cutting parameters.

The samples in as cold rolled and U-bend shape were prepared to a standard metallographic technique. First they were mounted in phenolic resin and then were manually wet polished with silicon carbide from 400 to 1200 mesh and followed by automatically polished with a magnetic polishing cloth. After which, the samples were etched with Villela's reagent for 15 minutes followed by air dried. The microstructure observation of the metallographic samples was carried out with an inverted type microscope, model Axiovert 100 MAT (Germany) equipped with image analysis software. The surface morphology of the U-bend sample with and without cold rolled process was analyzed using optical microscopy.
The micro hardness test was conducted according to Vicker's method using a micro-hardness tester, model Mitutoyo (Japan). The test was made at test forces of 0.98 N and 0.5 N with a loading time of 10 s. The hardness values of the U-bend samples were analyzed at the maximum bending region (Figure 11) whereas, at the transverse direction for the cold rolled samples.

Results and Discussion

Effect of Rolling on the microstructure

The strain hardening effects were characterized according to the percentage of dimension changes, in terms of its longitudinal or lateral increment to the rolling direction. An accurate measurement of the dimension changes was recorded using a digital vernier caliper, with decimals of 0.01 mm. **Table 2** shows the percent increment in length, %ΔL and increment in width, %ΔW of the cold rolled samples.

Table 2
Sample designations, length, width, percent increments, maximum bending loads and stresses.

CODE	%RT	Lo	Wo	ΔL	ΔW	P_{max}	σ_{max}
		mm	mm	%	%	N	MPa
SS-2.0	0	85.90	25.50	-	-	1857	11.6
SS-1.8	10	85.90	25.48	8.91	0.08	1875	13.0
SS-1.4	30	86.62	25.40	38.81	0.79	1814	16.2
SS-0.6	70	86.00	25.74	227.33	1.40	587	12.2

In general, the percentage of dimension changes, either in width or length, was directly depending on the degree of the cold deformation. The higher degree of the cold deformation was occurred, the superior the percentage of dimension changes. Moreover, the percent increment in width was found to be less than 2% for all samples and the percent increment in length was more than 8% and gradually increased to 227%. The result can be suggested that the increment in longitudinal direction produced a significant effect to the strain hardening of the cold rolled samples rather than lateral direction.

Figure 2 shows the microstructures of the as received material without being solution treatment, i.e annealing. The solution treated of 316L stainless steel was characterized by polygonal grains along with annealing twin boundaries **[6]**. For this research, the microstructure revealed polygonal grains consists of austenite phases, without annealing twin.

The cold rolling process caused to modify the microstructures of the 316L stainless steel due to plastic deformation. Plastic deformation caused grain hardening of the cold rolled steel (**Figure 3-5**), affecting its mechanical properties by increasing its resistance, flow stress, hardness and fragility; and by reducing its malleability, ductility and resistance to corrosion **[7]**. Plastic deformation also caused the formation of ferrite δ phase and the shrinkage of the austenite γ phases in the AISI 316L stainless steel and hardening of austenite phase became higher by reducing the thickness of the steel **[8]**. Martensite α' phase normally was not observed in AISI 316L steel after plastic deformation at room temperature **[9]**.

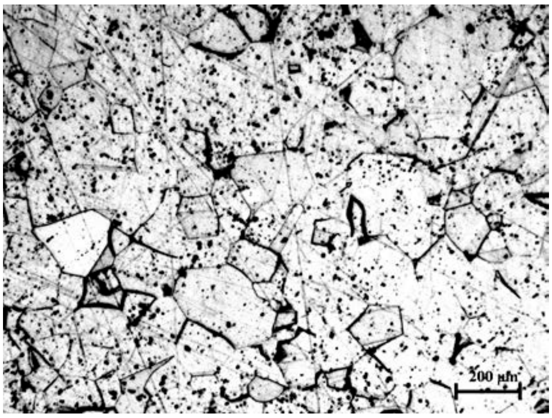


Figure 2. Optical micrograph of the as-received 316L stainless steel (magnifications of 200x).

When the steel heavily cold-rolled, up to 70%, much of the strain energy expended in the plastic deformation is stored in the material in the form of dislocation and other metal imperfection **[10]**. The strain energy extended in the plastic deformation contributes to the internal residual stresses.

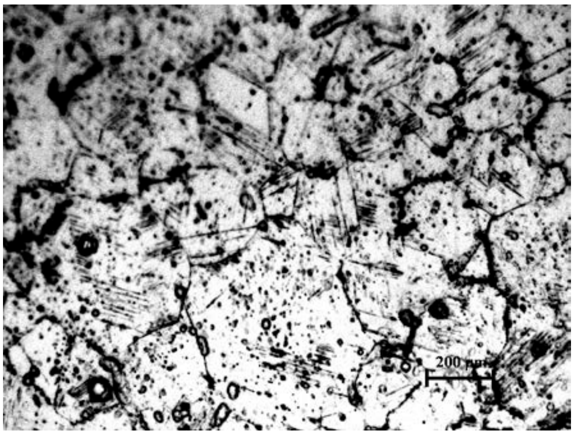


Figure 3. Optical micrograph of the cold rolled 316L stainless steel after 10 % thickness reduction (magnifications of 500x).

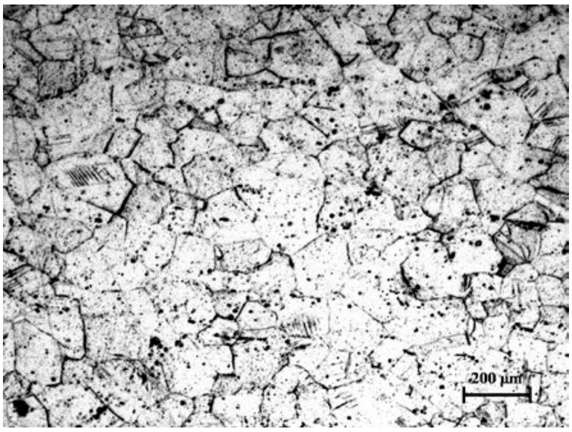


Figure 4. Optical micrograph of the cold rolled 316L stainless steel after 30% thickness reduction (magnifications of 200x).

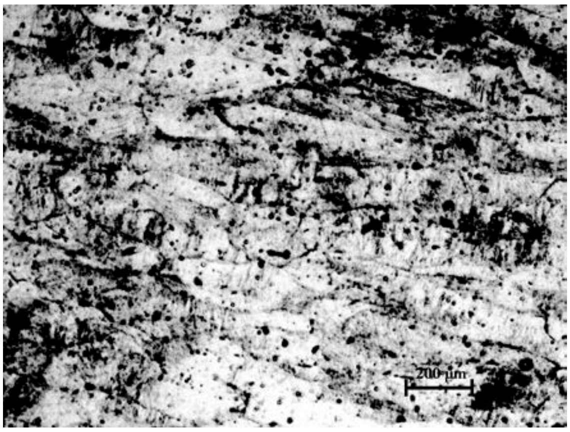


Figure 5. Optical micrograph of the cold rolled 316L stainless steel after 70% thickness reduction (magnifications of 200x).

The microstructures of 10 and 30% cold rolled samples (**Figure 4** and **5**) show the presence of twin boundaries. **Figure 6** shows the microstructure of 70% cold rolled sample indicated that a typical elongated and heavily dislocated grain structure. The microstructure consisting of dislocated tangles and banded structure. From these observations, the rolling process caused the microstructure modification of the cold rolled sample, by means of the grains hardening, from polygonal grains to heavily dislocated grains. Furthermore, the microstructures of cold rolled samples show the appearance of the δ phase along with the γ phase.

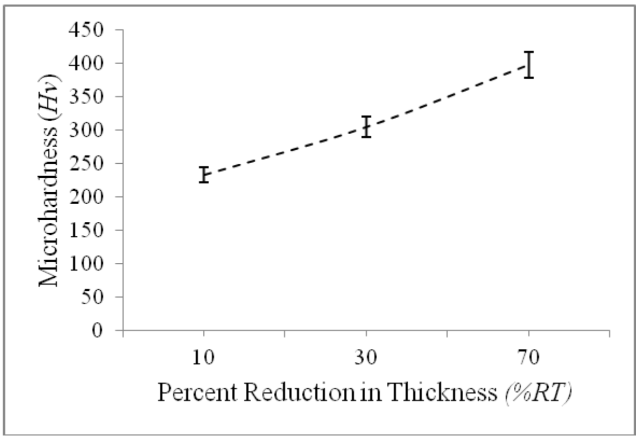


Figure 6. Microhardness values of the cold-rolled 316L stainless steel

In order to correlate the grain hardening with the mechanical properties, the micro-hardness test was performed. **Figure 7** shows the micro-hardness values of the cold rolled samples. The micro-hardness values were gradually increased from 233.3*Hv* to 398.3 *Hv* after thickness of the samples was reduced from 10 to 70%. As mentioned before, grain hardening of austenite phase becomes higher by reducing the thickness of the samples.

Effect of bending process on the microstructure

For the bending process, the cold rolled samples were slowly stressed in order to avoid misalignment of the U-shape and minimize internal defects. However, in bending sheets with a high width-thickness ratio, the cracks will occur near the center of the sheet when ductility is exhausted **[11]**.

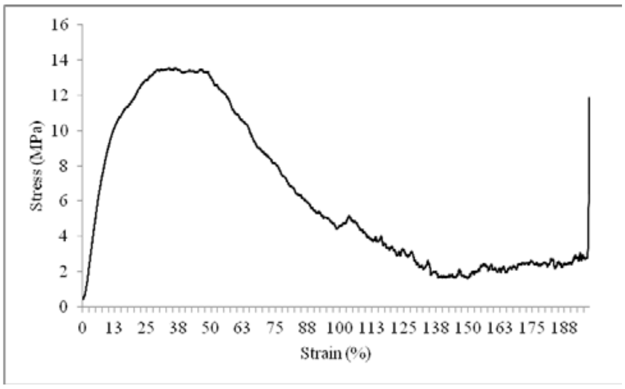


Figure 7. The stress-strain curve of the U-bend samples after 70% thickness reduction.

Figure 8 shows a stress-strain curve of the U-bend sample after 70% thickness reduction. From the curves, it shows that the maximum bending stress of 12.2 MPa need to be applied to produce the U-bend shape. The maximum value of bending stress and load of other samples were summarized in **Table 2**.

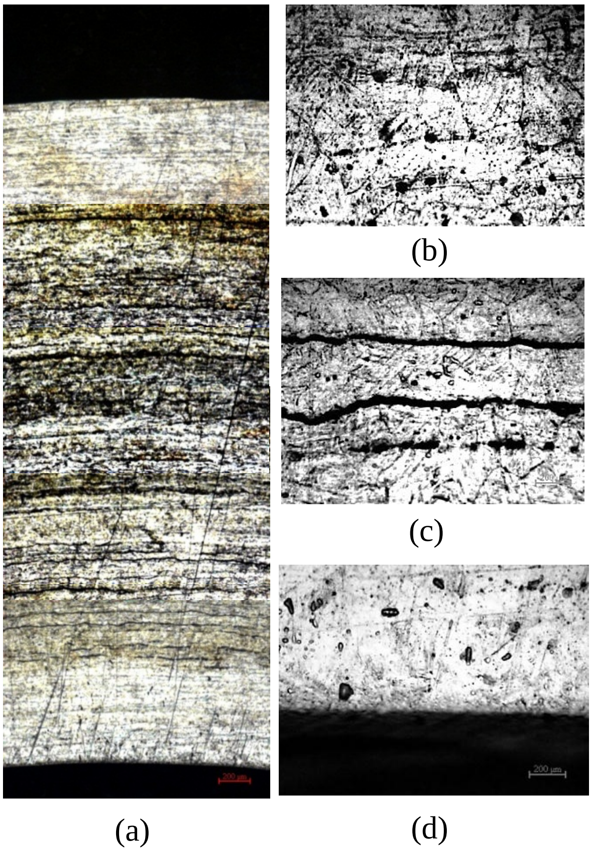


Figure 8 Surface morphology of the U-bend sample without cold rolled process at (a) the maximum bending point (magnifications of 100x); (b) top; (c) center and (d) bottom region (magnifications of 500x).

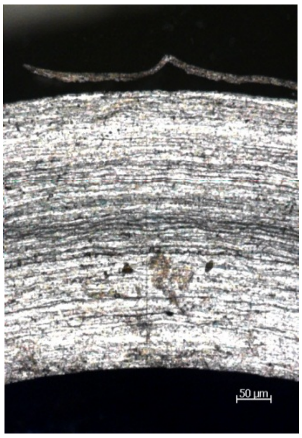
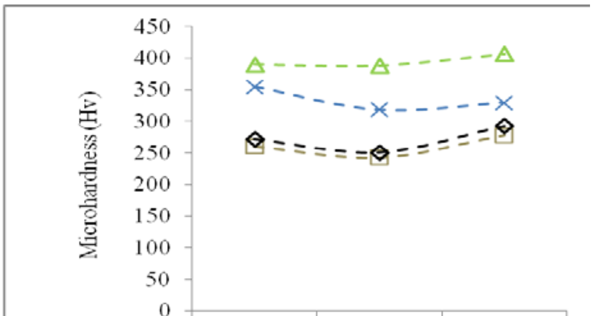


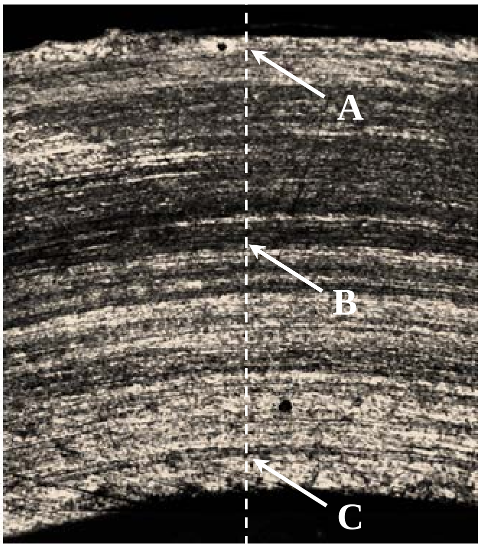
Figure 10 Surface morphology of the U-bend sample after 70% cold deformation at the maximum bending region (magnifications of 100x).

The surface morphology of the U-bend samples without cold rolled and with 70% cold deformation was shown in **Figure 8** and **9**. The surface morphology was indicated that the presence of inner cracks, parallel to the rolling direction, at the maximum bending point region. The inner cracks were clearly found at the center region for both samples, rather than at the top or bottom region. The inner crack occurred was supported by the previous statement, where the U-sample become more brittle after being cold rolled to 70% and have a large different between thickness and width with the ratio of 20:0.6.



	A	B	C
70% RT	390.4	388.4	406.7
30% RT	354.4	318.0	329.5
10% RT	260.1	242.4	278.0
0% RT	272.0	251.3	292.9

(a)



(b)

Figure 10. (a) Microhardness value of the U-bend samples after 10, 30 and 70% cold rolled at position A, B and C, and (b) A typical U-bend sample indicate the location of A, B and C region.

The microhardness values of U-bend samples in **Figure 11** were measured at the maximum bending region. The lowest hardness values were obtained at the center region (at point B) to all samples, due to formation of inner crack. The bottom region (at point C) shows the highest microhardness values for most of the samples, due to the region were slightly below the compressive surface.

Conclusion

From the analysis of the microstructure and mechanical properties of the rolling and bending process on the 316L stainless steel it can be concluded that: Rolling process caused the formation of austenite γ and ferrite δ phases. The grains hardening of the cold rolled samples were characterized with the presence of twin boundaries and dislocated grains. Additionally, the higher degree of plastic deformation permits to increase the hardness properties of the 316L stainless steel. Bending process caused the formation of the inner crack, parallel to rolling direction at the center region in the samples. The inner crack is responsible for reducing mechanical resistance, by means of the reductions of micro hardness values. The percent increment in longitudinal direction produced a significant effect to the strain hardening of the cold rolled samples rather than lateral direction.

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