Influence of Size on the Microstructure and Mechanical Properties of an AISI 304L Stainless Steel – A Comparison between Bulk and Fibers

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Abstract

This chapter deals with the study of mechanical properties and microstructural features of an AISI 304L stainless steel in two presentations: bulk and fiber form. Investigation was made in order to establish the relationship between the microstructure and the mechanical properties, as well as the influence of the fabrication process and the sample size. The microstructural and mechanical characterization of the products is presented and discussed. Different techniques, namely XRD, SEM and TEM, were used to assess the microstructure. The mechanical properties, specifically strength, Young’s modulus and elongation, were determined by tensile tests, Vickers microhardness and nanoindentation. The materials have been seen to possess different mechanical and microstructural mechanical properties, which are compared and analyzed.

Keywords

AISI 304L stainless steel; Fibers; Mechanical Properties; Microstructure

Introduction

Stainless steels are a group of engineering materials that have been used extensively owing to their properties, such as corrosion resistance and physical and mechanical properties
Among the various types of stainless steels, austenitic and ferrite types are ones of the most widely used. Ferritic stainless steels exhibit good ductility, formability, and moderately better yield strength relative to those of the austenitic grades, but the high-temperature strength is somewhat poor [3]. Nickel provides these properties by changing the crystal structure of steel to an austenitic fcc crystal structure at almost all temperatures. Conventional steel has a ferritic bcc crystal structure at ambient temperature. It is the addition of sufficient nickel - typically 8-10% - that imparts these unique properties. Metals with an austenitic fcc crystal structure are particularly tough and ductile. Aluminum, copper and nickel itself are good examples. Nickel is an austenite forming alloying, which can produce stainless steels with an austenitic structure stable at room temperature [4].

Austenitic stainless steels, particularly AISI 304L, usually have excellent corrosion resistance, good weldability and formability, good resistance to hydrogen embrittlement, in addition to high ductility and toughness. However, they have relatively low yield strength and hardness, reason why the application of austenitic stainless steel is mainly restricted to structural components, which require moderate flexural strength, torsional strength, impact toughness, and wear resistance. Multifarious surface treatments including nitriding, laser cladding, heat treatment [5], surface coating [6], and surface mechanical attrition treatment (SMAT) [7,8] have been adopted to enhance the mechanical properties of austenitic stainless steels. Some studies have also attempted to enhance the wear resistance of austenitic steels by grain refinement [9,10]. However, the high recrystallization temperature of stainless steel restricts the applicability of grain refinement because it generates large recrystallized grains rather than fine grains [11].

To solve the above, there are various strengthening mechanisms for austenitic stainless steels, such as grain refining, transformation strengthening and work hardening, converting them in materials widely used in engineering applications, such as in the manufacturing, nuclear, chemical, oil and petrochemical, and food industries, as well as the medical industry for biomedical implants [12,13]. Within the 300-series
austenitic stainless steels, the 304 grade is the most commonly used due to its superior low temperature toughness, as well as its corrosion resistance [14]. Recently, there has been an enormous amount of research addressing the improvement of the mechanical properties of austenitic stainless steel [10,15-18] without lowering corrosion resistance [19-22].

Stainless steels are in constant evolution to be used in different applications, being one of great impact the biomedical industry. However, the application in this industry has been limited, since one of the main alloying elements of stainless steels are nickel ions, which are considered to be an especially toxic species because it causes allergy and cancer [23]. The number of female persons affected by nickel allergy has doubled every one of the last few decades. Among the male population, this number has drastically increased only recently. High-nitrogen steels have been researched intensively in recent years [24]. At present, high-nitrogen stainless steels, especially high-nitrogen austenitic stainless steels have been the focus of attention, due to the cost of nickel and its toxicity.

Many experimental studies have focused on AISI 304 and AISI 304L stainless steels at elevated temperatures [25-27], under thermo-mechanical and cycle fatigue conditions [28-34], under creep conditions [35] and ductility loss of hydrogen-charged steel [36].

So far, a few research efforts have been performed on the comparison of the mechanical and microstructural properties of stainless steel bulk and fibers. Measurement of mechanical properties is a major part of the domain of materials characterization. Therefore, in the present work the mechanical properties and microstructural features of an AISI 304L stainless steel in two presentations, bulk and fibers, were systematically studied in order to establish the relationship between the microstructure and the mechanical properties.
Background on Bulk and Fiber Stainless Steel

Bulk stainless steels are commonly produced by hot rolling, followed by cold swaging and annealing processes. One great advantage of fibers and metallic wires is that they show very high-strength values and consistent properties, more so than any ceramic fibers. Conventional wire drawing methods are quite reasonable for producing steel wires with diameters all the way down to 100 μm, while diameters reaching to 10 μm or less can be obtained by the so-called Taylor process [37]. Steel fibers combine the flexibility of a traditional textile fiber with high-temperature resistance of steel. Stainless steel fibers are also resistant to mechanical stress, in particular to shearing stress, as opposed to glass, ceramic or carbon fibers, and they are resistant to corrosion. Stainless steel fibers have an excellent resistance to high temperatures.

The application fields of stainless steel fibers are the aircraft industry (embedded in an aluminum matrix), aerospace industry (along with boron, borsic and molybdenum fibers embedded in aluminum and titanium matrices), and rocket engines (within nickel alloys matrices) [37]. Steel wire is a common commercial reinforcement material, more for concrete than for metals or polymers. Steel fibers are commonly used as reinforcement in tires.

Fracture of metallic filaments differs in many respects from fracture of bulk samples. Particular manufacturing processes, such as drawing, melt spinning or crystallization from the vapor phase for fibers, that are needed to obtain their small lateral dimensions, may introduce specific defects and textures, and have influence on fracture behavior [38].

Analysis of Samples

The materials for this study were commercial AISI 304L stainless steel samples, both in bulk and fiber forms. Different characterization techniques are available for the analysis of materials, ranging from the most common and accessible techniques to the most sophisticated and, sometimes, not
available to everyone. Some of these techniques are described and used below.

**Chemical Analysis**

There are several qualitative and quantitative techniques to determine the chemical composition of materials. They range from wet chemical methods to modern instrumental methods. Among the latter are atomic absorption spectrometry, flame atomic absorption spectrometry, inductively coupled plasma optical emission spectrometry (ICP-OES), inductively coupled plasma mass spectrometry, and CHNS-O elemental analyzer. These techniques are applied depending on the range of concentrations of samples. The ICP-OES is an analytical technique based on the principles of atomic spectroscopy, which is useful to determine the elemental composition in materials of different nature; its concentration range is from major to trace (parts per billion) [39].

In this investigation, the chemical composition of the stainless steel samples (bulk and fibers) was acquired by inductively coupled plasma optical emission spectroscopy using a Thermo Scientific iCAP 6500 spectrometer, as well as by an EA 1110 CHNS-O elemental analyzer from CE Instruments. Table 1 summarizes the results of the chemical analysis, where it can be seen that the composition matches well to the AISI 304L standard [40].

**Table 1:** Chemical composition of the bulk and fiber specimens of 304L austenitic stainless steel (wt%).

| Sample  | C   | Si  | Mn  | Cr    | Ni   | P   | S   | Fe          |
|---------|-----|-----|-----|-------|------|-----|-----|-------------|
| Bulk    | 0.023 | 0.278 | 1.488 | 18.163 | 8.214 | 0.026 | 0.024 | Balance     |
| Fiber   | 0.029 | 0.389 | 1.406 | 19.756 | 11.216 | 0.013 | 0.005 | Balance     |
| ASTM A240 | 0.030 | 0.750 | 2.000 | 18.000-20.000 | 8.000-12.000 | 0.045 | 0.030 | Balance     |

**Microstructural Characterization**

The microstructural study of samples can be performed from simple and economical techniques like optical microscopy to
complex and expensive techniques like scanning electron microscopy, transmission electron microscopy, high-resolution transmission electron microscopy, and scanning transmission electron microscopy.

**Optical Microscopy:**

Optical microscopy (OM) commonly uses visible light and a lens system to generate magnified images of a sample. OM is a nondestructive and real-time imaging technique, being one of the most powerful and versatile investigation techniques in material sciences. It is the simplest technique for the superficial and morphological characterization, which allows to determine fairly quickly several features about the microstructure of analyzed samples.

For the analysis of the fracture surface and porosity measurements of the 304L stainless steel samples, an Olympus PMG3 optical microscope, along with an Image-Pro Plus image analyzer, were employed. These results will be presented below.

**Scanning Electron Microscopy:**

Due to the limitations that optical microscopy presents, electron microscopy takes advantage of much smaller wavelengths, in terms of qualitative and quantitative analysis on materials, to gather comprehensive information on details and defects in smaller and specific areas. The capabilities of electron microscopy are restricted to the resolution limits of the equipment currently available.

Scanning electron microscopy (SEM) is a technique for the observation and characterization of materials from the micro (μm)- to the nano (nm)-scales. It is one of the most versatile techniques available for the examination and analysis of the microstructural features of solid objects, which takes advantage of the use of electrons to form high-resolution images with highly detailed information about the sample [41]. The re-emitted particles produced by the electron beam can be analyzed by different detectors, allowing the acquisition of information
related to the morphology, surface defects, phase distribution, depth of field, and chemical composition. There are scanning electron microscopes equipped with a field emission electron gun (FESEM), which have a better resolution, higher brightness, and reduced noise level than those of conventional ones.

In this work, the microstructural features and fracture of the bulk and fiber AISI 304L stainless steel were characterized by FESEM using a JEOL JSM7401F microscope. Figure 1 shows secondary electron SEM images of an as-received 304L stainless steel fiber. Fibers appear smooth and are approximately circular; a circular cross section was considered for stress calculation. They do not apparently present surface defects, except some longitudinal striations along their axis, produced by the manufacturing process. An average diameter of 45.29 ± 0.30 μm was determined, as evidenced in Figure 1a.

![Figure 1: SE-SEM micrograph of individual 304L stainless steel fibers: (a) longitudinal and (b) cross-section.](image)

The SEM observations revealed that both fibers (Figure 2a) and bulk (Figure 2d) specimens possess the same polygonal microstructure, similar to the cold-worked AISI 304L condition, with irregular austenite grains and dispersed carbide particles. The cold work in austenitic stainless steels induces the martensitic transformation, and different martensite morphology can be formed. In Figure 2, slip bands in the austenite matrix are observed, which is indicative of the presence of martensite generated during the cold-work process. Due to the micrometer size of fibers, the micrographs are presented at different magnifications in comparison with those of the bulk material. It
is important to notice that there exists a significant difference in grain size between both materials; a reduction of diameter during the fiber processing leads to the refinement of grain size.

Figure 2: SE-SEM micrographs of the AISI 304L samples: (a) cross and (b) longitudinal sections of fibers; (c) cross and (d) longitudinal sections of bulk material.

Transmission Electron Microscopy:

Transmission electron microscopy (TEM) is a technique that uses electrons passing through the sample to generate an image [42]. The sample must have an ultrathin thickness of ~100 nm for an appropriate analysis; for this task, techniques such as electropolishing, ultramicrotomy, and focused ion beam are used. The interaction of the electron beam with the material produces diffraction of the transmitted electrons in a coherent way to the crystalline planes of the material. Besides these capabilities provided by TEM, a chemical characterization of samples is possible to do with the use of energy-dispersive spectroscopy (EDS) coupled to the transmission electron microscope. The EDS technique detects X-rays emitted from the
material to determine the elemental composition of the analyzed volume. The screened X-ray energy is characteristic of the element from which it was emitted due to the fact that each element possesses a unique atomic structure, obtaining a unique set of peaks on its X-ray spectrum.

In the present study, phase and precipitate composition of the bulk and fiber samples were identified by TEM using a JEOL-JEM2200FS microscope. Thin foils from the specimens were prepared by focused ion beam (FIB). There are several strengthening mechanisms for austenitic stainless steels, such as grain refining and work hardening. The examination revealed a micrometer and sub-micrometer microstructure (Figure 3a). In addition, the presence of nanosized precipitates was found (Figure 3b). According to the EDS-TEM results presented at the bottom of Figure 3, the formation of nanosized carbides during the fiber processing is suggested. The presence of nanocrystalline phases in the fibers can influence their mechanical properties.

Figure 3: TEM micrographs: (a) bright field; and (b) Z-contrast images of a fiber sample showing the presence of nanoscale precipitates.
Structural Characterization

There are diverse techniques for the structural analysis of samples, being X-ray diffraction (XRD) an important and versatile tool used in materials science for the characterization of materials. It is a rapid analytical technique mainly used for the phase identification of a crystalline material, providing information on the unit cell [43]. XRD is able to give immediate information on the phase composition and crystalline features after synthesizing and processing a sample. The technique is very sensitive to composition, but additions around 1% are difficult to identify. In this research, XRD measurements were performed on the samples for the phase identification, using a Panalytical X’Pert PRO diffractometer with Cu Kα radiation (\(\lambda = 0.15406\ \text{nm}\)). The XRD patterns were indexed with X’Pert HighScore Plus software containing the PDF-2 files database.

XRD patterns of the studied samples are plotted in Figure 4 for the 2θ region from 35° to 125°, with the Bragg reflections indexed. They revealed that both bulk and fiber samples have the same crystalline structure. During plastic deformation of austenitic stainless steels at room temperature, the martensitic transformation occurs from the austenite phase. The structure observed in the diffraction peaks consists of a mixture of bcc and fcc phases, corresponding to the characteristic AISI 304L \(\alpha\)-Fe (martensite) and \(\gamma\)-Fe (austenite) phases, respectively. Nevertheless, the XRD patterns of AISI 304L samples in different states present a remarkable difference in intensity. The peaks of the bulk sample are sharp, while those of the fiber pattern exhibit a significant shortening that indicates a crystal size refinement. The diameter size reduction of fibers produced by cold working causes lower intensity of austenitic peaks (\(\gamma\)-Fe) in comparison to the bulk sample, while diffraction peaks of \(\alpha\)-Fe phase cannot be easily found due to the fine crystal size. The crystal size and microstrains were determined by the Williamson-Hall method. The found values of crystal size were 51.5 \(\mu\text{m}\) and 2.32 \(\mu\text{m}\) for the bulk and fiber samples, respectively. The microstrains of fibers (0.232%) exceeds the value of the bulk sample (0.181%).
lattice parameters for the bulk sample are $a_{\alpha-Fe} = 0.287$ nm and $a_{\gamma-Fe} = 0.359$ nm, while for the fiber sample only the peaks corresponding to the $\gamma$-Fe phase were observed, with a lattice parameter of $a_{\gamma-Fe} = 0.358$ nm. A significant lattice mismatch was not found in both samples. On the other hand, the variation in the relative intensities of the main diffraction peaks of the bulk sample in comparison to the fiber 0°, fiber 90° and fiber spinning patterns, suggests the presence of crystallographic texture due to the processing route of the fibers.

![XRD patterns](image)

**Figure 4:** XRD patterns of the bulk and fiber samples.

**Mechanical Properties**

The mechanical properties of a material are those properties that involve a reaction to an applied load. Different techniques are used for the evaluation of the mechanical behavior of materials, such as tensile, compression, bending, impact, fatigue, creep, and hardness tests, among others. The mechanical properties of a
material are not constants and often change as a function of temperature, rate of loading, and other external conditions. There is often significant variability in the values obtained when measuring mechanical properties. A seemingly identical test specimen from the same lot of material will often produce considerably different results. Therefore, multiple tests are commonly conducted to determine the mechanical properties, and the values reported are the average value or calculated statistical minimum value. The mechanical properties can be evaluated at different scales, going from the bulk scale, through the microscale and to the nanoscale.

**Tensile Tests:**

Regarding the bulk specimens, tension tests were carried out at room temperature, according to the ASTM E8 / E8M standard [44]; an Instron 4469 universal testing machine with a load cell of 50 kN was used. A strain rate of $4.0 \times 10^{-3}$ s$^{-1}$ and a specimen gauge length of 30 mm were used. Yield strength, ultimate tensile strength and fracture strain of the specimens were calculated from the stress-strain curves acquired; an extensometer and a linear regression were used for obtaining the Young’s modulus.

In the case of the 304L stainless steel fibers, they were tested individually in a universal fiber tester developed originally by Bunsell et al. [45], which is capable of conducting tensile, relaxation, creep and fatigue tests on very small diameter filaments. The tests were performed at room temperature using a load cell of 250 g calibrated from 0 to 100 g, with a 0.01 g of precision. Before testing, the fiber specimens were glued to card supports so as to give a gauge length of 30 mm. The card protected the fibers from the machine grips. The tests were conducted at a strain rate of $4.0 \times 10^{-3}$ s$^{-1}$. The data acquisition used a PC linked to the fiber tester via a National Instrument interface card and WinATS 6.2 software from Sysma. In order to normalize the stress, the diameter of each fiber was systematically measured before each test by a Mitutoyo LSM-500S laser device, with an accuracy of 0.01 μm. The calibration
of this device was performed using some fibers whose diameter was previously measured by SEM.

The engineering stress-strain curves of both the bulk and fiber specimens are shown in Figure 5. According to Table 2, it can be seen that the fiber samples show lower tensile mechanical properties than those of the bulk samples. The parabolic shape of the bulk specimen curve indicates that strain hardening occurs throughout the duration of the stress application, but such an amount of strain hardening for a given increment of stress decreases as stress increases. Concerning the fibers, instead of a parabolic-shaped curve, the stress increased monotonically up to failure, which means that there is always a strain hardening of the fiber structure. It is clear that the bulk mechanical properties exceed those of the fibers.

An improvement of yield stress ($\sigma_y$) and Young’s modulus (E) in the fibers was expected due to grain refinement in comparison to the bulk sample, however the result was opposite to that expected. During the cold work, crystalline defects like dislocations and porosity increase with the degree of deformation and decrease the mechanical properties. In addition, the sample sizes (diameter of 45.29 μm for fibers and 6.41 mm for bulk) affect the results of mechanical tests. The coupled effect of crystalline defects and tested area size of samples is evident in the reduction of mechanical properties of fibers evaluated in macro-scale in comparison to the bulk sample.
Figure 5: Stress-strain curves of the bulk and fiber samples.

Table 2: Mechanical properties of 304L stainless steel samples.

| Sample      | \( \sigma_y \) (MPa) | \( \sigma_{\text{max}} \) (MPa) | \( \varepsilon \) (%) | \( E \) (GPa) | HV  |
|-------------|-----------------------|-------------------|-----------------|----------|-----|
|             |                       |                   |                 |          | Long-section | Cross-section |
| Bulk        | 536 ± 5.2             | 720.73 ± 4.4      | 49.24 ± 3.1    | 107.73 ± 12.5 | 273.53 ± 5.1 | 256.53 ± 7.2 |
| Fiber       | 344 ± 4.8             | 527.53 ± 5.0      | 16.33 ± 2.8    | 97.07 ± 9.3  | 151.73 ± 9.1 | 169.12 ± 2.0 |

Figure 6 presents optical microscopy images of the samples after the tension tests. Necking and neck propagation were observed for both materials, which are associated with ductile materials (Figures 6a,c). Ductile fractures are characterized by tearing of metal along with appreciable gross plastic deformation. Ductile tensile fractures in most materials have a gray and fibrous appearance [22]. The quantity and size of pores after tensile tests can be seen in Figures 6b,d for the fiber and bulk specimens, respectively; in the case of fibers, the fracture surface contains...
many honeycomb-like deep dimples, distributed homogeneously throughout the whole surface.

Figure 6: Tensile fracture surface morphologies of (a) and (b) fiber, and (c) and (d) bulk material.

The porosity percentage of the studied materials was determined by optical microscopy; the measurements were conducted on the polished cross-section of samples (Figure 7). The bulk sample porosity was found to be 2.13%, while the fiber porosity was 5.84%. The mean pore diameter was 1.46 ± 0.21 μm and 4.21 ± 0.88 μm for bulk and fiber samples, respectively. Hence, it is evident that a greater volume fraction of porosity has a detrimental effect on the mechanical response of fibers, in comparison to the bulk condition. Porosity is known to have a significant effect on the mechanical properties of metals, in particular on the yield stress, ultimate tensile strength and fracture strain, as shown by the results in Table 2. In the case of the Young’s modulus, this is a material intrinsic property; nevertheless, in the present work a decrease in Young’s modulus was found due to the porosity, especially for fibers. A similar behavior was reported by Qiao et al. [46] for 316L stainless steel.
fibers, where the Young’s modulus decreased with increasing sample porosity. Furthermore, Chawla and Deng [47] found that increasing density results in an increase in Young’s modulus for sintered steels.

Figure 7: Optical microscopy images showing the measured diameter size of pores, represented by open red circles: (a) fiber and (b) bulk specimens.

Vickers Microhardness and Nanoindentation:

Hardness of both kind of samples was measured using the Vickers microhardness method on the longitudinal and cross sections of polished samples. A Future-Tech FM microhardness tester was employed under a load of 200 gf and a dwell time of 15 s. Nanoindentation tests were carried out by an Agilent Nano Indenter G200 under the G-series XP cycles interactive indentation mode, using a diamond Berkovich indenter tip with a radius of 20 nm, strain rate target of 0.05 s\(^{-1}\), harmonic displacement target of 1 nm and a frequency target of 75 Hz. The fibers were vertically and horizontally embedded in an epoxy resin and cured in a plastic mold; after curing, the fibers were hand polished in order to provide a smooth exposed surface and measurements were performed in their cross and longitudinal sections.

The results of hardness are presented in Table 2, which shows that the hardness of the bulk samples is higher than that of fibers. This lower microhardness of fibers can be due to their higher porosity; that is why nanoindentation measurements on the cross section were performed in order to determine hardness and Young’s modulus of bulk and fiber specimens. Concerning the
hardness measurements by nanoindentation, the bulk material presented a value of 4.19 GPa, while the fibers presented a value of 4.97 GPa; in both cases these results are significantly greater than those reported in Table 2: 2.52 GPa (256.53 HV) and 1.66 GPa (169.12 HV) for bulk and fibers, respectively. This can be explained by the fact that the nanoindentation measurements were carried out in flaw-free small regions, namely porosity-free, while Vickers microhardness measurements were performed over large regions that might contain porosity. In reference to the Young’s modulus, the values found by nanoindentation for the bulk and fiber specimens were 181.08 and 198.04 GPa, respectively, which are close to that reported for the AISI 304L stainless steel (193 GPa) [14]. According to Young’s modulus results obtained by tensile tests (Table 2) and nanoindentation, the values for bulk material are similar by both techniques, but in the case of fibers, the value is less for tensile tests (115.93 GPa); this difference can be again explained by the large number of flaws that samples contain throughout their longitudinal section, especially in the case of fibers. The hardness and the Young’s modulus measured by nanoindentation are higher because the measurements were performed over defect-free areas.

The higher values in fiber properties measured by nanoindentation yield evidence that deformation of fibers during the cold work induces greater material strengthening, making it stiffer by reducing their deformation ability without reaching a completely brittle material.

**Conclusions**

In this work, tensile, microhardness and nanoindentation tests were done on a 304L stainless steel in the form of bulk and fibers, in order to evaluate and compare the influence of the microstructure on their mechanical properties. Both presentations showed a similar microstructure and tensile fracture morphology. Tensile and hardness tests showed that the bulk sample has higher values of yield stress, ultimate tensile stress, Young’s modulus, elongation and hardness than fibers, as a result of their porosity and pore size. It was observed that the macroscopic (tensile tests) and microscopic (microhardness
tests) properties of the fibers are sensitive to these defects generated during the material manufacturing process.

A very small volume of material could be tested throughout nanoindentation testing. The superior mechanical properties of bulk and fibers obtained by nanoindentation, in comparison with those obtained by tensile tests and microhardness, are due to the capability of nanoindentation of performing measurements on a small and porosity-free area. With this technique, higher values of hardness and Young’s modulus on the fiber form were reached in comparison to the bulk form. These results were expected due to the greater plastic deformation induced in fibers during their processing. On the other hand, opposite results were obtained by microhardness and tensile tests, as greater pore density in fibers led to a reduction in the mechanical properties of the researched material.

Smaller crystal and grain size of fibers, in comparison to bulk samples, had a significant effect on their mechanical behavior measured by nanoindentation.

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