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AN ANALYSIS UPON MICROSTRUCTURAL DEVELOPMENTS AND MECHANICAL PROPERTIES OFAISI 304L STAINLESS STEEL

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An Analysis upon Microstructural Developments and Mechanical Properties Ofaisi 304L Stainless Steel

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Abstract – In this 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 among microstructure, mechanical properties, manufacturing process and effect on sample size. The microstructure was analyzed by XRD, SEM and TEM techniques. The strength, Young's modulus and elongation of the samples were determined by tensile tests, while the hardness was measured by Vickers microhardness and nanoindentation tests. The materials have been observed to possess different mechanical and microstructural properties, which are compared and discussed.

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.

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INTRODUCTION

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 in the annealed state. 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. 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. Recently there has been an enormous amount of research addressing the improvement of the mechanical properties of austenitic stainless steel without lowering corrosion resistance. Many experimental studies have focused on AISI 304 and AISI 304L at elevated temperatures, under thermo-mechanical and cycle fatigue conditions, under creep conditions and ductility loss of hydrogen-charged 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. Steel fiber combines 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 matrix), and rocket engines (within nickel alloys matrices). 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.

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.

It is noteworthy that the 304L stainless steel fibers were tested individually in a Universal Fiber Tester, capable of conducting tensile, relaxation, creep and fatigue tests on very small diameter filaments, with a resolution of 0.01 g.

AISI type 304L austenitic stainless steel (304L SS) is widely used in power, chemical, petrochemical and nuclear industries. 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. Semi-solid processing is one such promising alternative to conventional manufacturing techniques, 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, such as aluminum and magnesium alloys. The suitability of this technology has also been investigated for producing some components in laboratory scale from the ferrous alloys, such as X210Cr and bearing steels. Therefore, adoption of this technology for other grades of steels has drawn worldwide attention. The major constraints in semi-solid processing of steels arise * Corresponding author: deepsaroj@igcar.gov.in (Dipti Samantaray) Published online at http://journal.sapub.org/ijmee Copyright © 2013 Scientific & Academic Publishing. All Rights Reserved from high melting point of these alloys, narrow semi-solid temperature range, and phase transformation during melting and solidification. 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.

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. 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. There are several relationships available to correlate the hardness with the yield strength and ultimate tensile strength of materials. 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 1. 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 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 describe 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 1 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.

	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

Table 1. Mechanical properties of the conventional and semi-solid processed specimens.

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. As delta-ferrite has a bcc structure, 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, most significantly by pining the dislocations and reducing their mobility. 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.

TENSILE AND COMPRESSIVE PROPERTIES OF AISI 304L STAINLESS STEEL

Equal channel angular pressing (ECAP) is presently one of the most promising techniques which can produce ultra-fine grained (UFG) materials (grain size in the range of 10–1000 nm) through the process of simple shear by pressing a sample through a die with two intersecting channels, equal in cross-section.

The sample is simply pressed through the channel and a shear strain is introduced to the sample when it passes through the bending point of the channel . Repetitive pressing is feasible as the sample cross-section remains unchanged. A high total strain is then achieved during a process of multiple-pass pressing. Between each successive pressing, it is possible to select one of the four distinct routes, designated as routes A, BA, BC and C, respectively, in which the sample is rotated 0◦, 90◦ and 180◦ along its longitudinal axis, respectively. However, the routes designated as routes BA and BC refer to a rotation of 90◦ in the opposite sense and in the same sense between consec- utive passes, respectively. The four processing routes were examined earlier with reference to the macroscopic distortions occurring on the *X*, *Y* and *Z* planes of the work-piece. Therefore, the strength of ECAP resides in its versatility and ability to reproduce different microstructures and textures via numerous conventional processing methods such as rolling and drawing, just by changing the strain path. Furthermore, it is found that the ECAP process yields a lamellar microstructure when the orientation of the billet is not changed after each pass and the development of combined ECAP routes involving orthogonal deformation paths contributes to achievement of smaller grains and a more equiaxed microstructure. The advantage of this processing technique has been manifested by the improved properties of materials, including mechanical properties and physical properties. Some promising improvement of strength in different materials, for example, copper, Al-alloys, pure Ti, Cu-based alloys, low-carbon steel, processed by ECAP has been achieved recently.

The austenitic stainless steel is one of the important structural materials and has many applications in industry mainly because of its excellent corrosion resistance. However, its low yield strength is often a major drawback. Since severe plastic deformation (SPD) techniques such as ECAP or high-pressure torsion (HPT) can effectively improve the yield strength of materials, it is reasonable to consider applying such a technique to austenitic stainless steel. Belyakov et al. investigated initial grain size effect on the evolution of sub-microcrystalline structure during multiple compression of a 304 stainless steel at 600 ◦C. They found that the ultra-fine grains were developed as a result of a continuous increase in the misorientations between the subgrains evolved during deformation. In the samples with grain size smaller than 3.5 μm, the fraction of strain-induced high-angle boundaries rapidly increases to more than 60% with a strain of about 1.5. However, their fraction does not exceed 20% at a strain of 1.5 in the sample with grain size of 15μm. Besides, they investigated the static restoration mechanisms operating during annealing in a 304 stainless steel at a straininduced submicron grain microstructure. Yapici et al. studied the microstructure refinement and deformation twinning in 316L stainless steel subjected to ECAP. Recently, Huang et al. successfully obtained bulk nanocrystalline grain structures in ultra-low carbon stainless steel by means of ECAP at room temperature. TEM investigations indicated that two types of nanostructures were formed: nanocrystalline strain-induced martensite with a mean grain size of 74 nm and nanocrystalline austenite with a size of 31 nm characterized by dense deformation twins. This result suggests that low stacking fault energy is exceptionally profitable for producing nanocrystalline materials by ECAP. Although there are some reports on the microstructure evolution of the ECAPed stainless steel, the corresponding mechanical properties of the ECAPed stainless steel with a relatively fine microstructure were not adequately explored. In the present work, the compressive and tensile properties of AISI 304L stainless steel subjected toECAPwere systematically investigated in order to establish the relations among the number of ECAP passes, microstructure, mechanical properties and the deformation and fracture modes. Furthermore, the anisotropic compressive mechanical properties of the AISI 304L stainless steel were discussed for a better understanding of the ECAP mechanism.

EXPERIMENTAL SECTION

The materials for this study were commercial AISI 304L stainless steel samples, both in bulk and fiber states. The chemical composition of the researched materials was performed by inductively coupled plasma atomic emission spectroscopy using an iCAP 6500 Thermo Electron spectrometer (Chihuahua, México), as well as an EA 1110 CHNS-O Elemental Analyzer (Chihuahua, México) from CE Instruments. The microstructural features and fracture of both kinds of materials were characterized by scanning electron microscopy (SEM, Chihuahua, México), using a JEOL JSM7401F microscope (Chihuahua, México). Phase and precipitate composition were identified by transmission electron microscopy (TEM, Chihuahua, México) using a JEOL-JEM2200FS microscope (Chihuahua, México). X-ray diffraction (XRD, Chihuahua, México) measurements were performed on the specimens for phase identification, using a Panalytical X'Pert PRO diffractometer with Cu Kα radiation (λ = 0.15406 nm). The XRD patterns were indexed with X'Pert HighScore Plus software containing PDF-2 files database. Single fibers were subjected to tensile tests at room temperature using a universal fiber tester developed originally by Bunsell *et al.* , equipped with a load cell of 250 g calibrated from 0 to 100 g, with a 0.01 g of precision. 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 $4.0 \times 10^{-3} \cdot s^{-1}$. 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 (Chihuahua, México), with an accuracy of 0.01 µm. The calibration of this device was performed using some fibers whose diameter was measured by SEM. Tension tests on bulk specimens were carried out at room temperature, according to the ASTM-E8M standard; an Instron 4469 universal testing machine with a load cell of 50 kN was used. A strain rate of $4.0 \times 10^{-3} \cdot s^{-1}$ 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 obtained; an extensometer and a linear regression were used for obtaining the Young's modulus. For porosity measurements, an optical microscope (OM, Chihuahua, México) Olympus PMG3 and an Image-Pro Plus image analyzer (Chihuahua, México) were used. Fracture analysis for fibers was carried out through SEM observations, while for bulk materials it was carried out through OM. Hardness of both specimens was measured using the Vickers microhardness method on the longitudinal and cross sections of polished samples. Nanoindentation tests were carried out by an Agilent Nano Indenter G200 (Chihuahua, México), using 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.

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.

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, maximal stress, Young's modulus than fibers, elongation and hardness, 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.

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