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PHYSICAL CHARACTERIZATION OF AUSTENITIC STAINLESS STEELS AISI 304 AND AISI 348 L*

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ABSTRACT

The study of radiation damages in metals and metallic alloys used as structural materials in nuclear reactors has a strategic meaning to the nuclear technology because it treats of performance of these materials in conditions that simulate the conditions of work in power reactors. Then it becomes necessary to know the essential physical properties of these materials, properties that are sensitive to the microstructural changes that occurred during the irradiation. The purpose of this work is to characterize, initially, some pre-irradiation properties of the stainless steels AISI 304 and AISI 348 L*, such as mechanical (stress-strain and microhardness) and electrical (resistivity). The AISI 348 L* has been studied for use as fuel cladding material. Both materials will be tested after irradiation in the IEA-R1 core and their properties will be compared with those in the pre-irradiated condition. The morphology of the fractured zones after tensile tests was observed using SEM (scanning electron microscopy)

1. INTRODUCTION

The materials used in nuclear reactors in addition to being subject to aggressive conditions such as corrosive environments, mechanical strain, high thermal gradients, etc. support an additional condition of high energy particles neutron, which cause changes in its crystalline structure, thereby altering their mechanical properties. H. Ullmaier and W. Schilling [1] review some major aspects related to radiation damage of metallic materials, involving safety and economy in nuclear reactors. Thus, it is essential to gain the most possible knowledge of the mechanisms of radiation damage and use this information to develop alloys more resistant to radiation.

From this point of view, the Fuel Engineering Group of the IPEN-CNEN/SP has adopted a program of characterization of austenitic stainless steels for applications in PWR reactors. Since 1990’s years the study of radiation damage in the reactor environment is one of the stages of this program, beyond the effect of helium gas, which is created in structural materials by reaction (n,α), and because of its extremely low solubility [2], tends to agglomerate into bubbles at high temperatures, causing embrittlement leading to premature rupture of the material [3].

The structural materials to be tested in the first use of the CIMAT (*Cápsula de Irradiação de Materiais*) irradiation device will be the AISI 304 and AISI 348 L*. Both steels AISI 348 L* and AISI 304 can be used as structural materials of reactor components. The difference in their compositions is the addition of Nb and N in AISI 348 L*, that gives increased mechanical strength and a decrease in intergranular corrosion. The AISI 304 has a normal content of C (0.058% by weight) which contributes to the precipitation of $Cr_{23}C_6$ in the grain boundary, showing a strong intergranular corrosion, which is extremely harmful from the structural point of view. Hence the need to produce a material with good mechanical properties and to decrease the precipitation of undesirable carbides in the grain boundary. Initially, it was chosen a material with low C, which would reduce the precipitation of $Cr_{23}C_6$. The decrease in content of C would carry a significant loss of mechanical strength, so it was then proposed a new material which would have a low content of C (for the reduction of $Cr_{23}C_6$ precipitates), and would have a strength greater than steel AISI 304 . The adding of Nb (Nb = 10% C) in solid solution gives a increased mechanical strength. Moreover, N is added to compensate, further, the loss of mechanical strength due to the decrease in content of C, as described previously. The steel AISI 348 L* as a modified steel, besides its low precipitation of carbides of type $Cr_{23}C_6$ has, likely a mechanical strength greater than steel AISI 348 L (without the addition of N).

The objective of this work is to characterize the stainless steel AISI 348 L*, that has been studied for use as fuel cladding material in comparison with stainless steel AISI 304. Experimental results are presented for the materials in the non-irradiated condition. The following characteristics were obtained: mechanical properties (stress-strain and microhardness) and electrical property (resistivity). The morphology of the fracture zone of test specimen after the tensile test was verified by scanning electron microscopy.

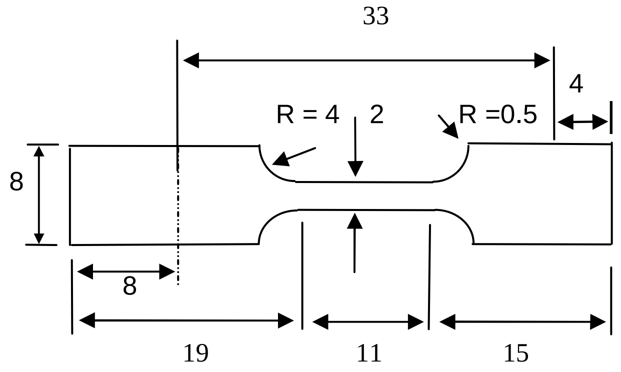
2. EXPERIMENTAL PROCEDURE

The chemical composition of the austenitic stainless steel AISI 304 and 348 L* are showed in Table 1.

Table 1. Chemical composition of steels (% by weight) [4]

Element	AISI 304	AISI 348 L*
C	0.058	0.029
Si	1.01	0.38
Mn	0.7	1.74
P	0.016	0.003
S	<0.01	0.003
Cr	18.80	17.81
Ni	8.46	10.12
Nb + Ta		0.36
N		0.069
Co		0.02

The original material was received in the form of rectangular wrought bars in dimensions (4x6x20) cm, with rough finishing. These bars were cut in the longitudinal direction, taking samples of approximately 1 mm each, which were rolled to a thickness of 100 µm, from these bars, sheets of 17 mm wide and 60 mm in length. During the rolling, to relieve the internal stresses due to material hardening, intermediate annealing were done to 900 ºC for 3 hours in dynamic vacuum of 10^{-5} Torr. Then samples were cut by electroerosion (avoiding in this way, the introduction of deformation in the material) and the test specimen were submitted to a initial heat treatment of 900 ºC for 3 hours in dynamic vacuum of 10^{-5} Torr. The specimens were submitted to tensile tests, microhardness, electrical resistivity and scanning electron microscopy (SEM) in the region of fracture.



Dimensions: mm
Thickness: µm

Figure 1. Outline of the test specimen used in this work

Tensile test. The schematic drawing of the specimens used in the tensile test can be seen in Fig. 1. The effective length of specimen was considered as 11 mm. The tests were performed on a machine Instron, model 5567, with the load cell of 1 kN. The specimen were load until the rupture.

Microhardness. Measures of microhardness were performed in a microdurometer Otto Wolpert Werke, with load of 100 g. To this purpose, the specimens, embedded in epoxy resin, were smoothed in the sequence, using sandpaper from 240 to 600. Then, they were polished by diamond paste, in the sequence of 6, 3 and 1 um. After the polishing they were attacked in an electrolyte solution of oxalic acid (10% by weight) for 1.5 min, with an electric current of 1 A/cm^2.

Electrical Resistivity. The specimen used in the electrical resistivity is the same as showed in the Fig. 1. The four wire method was employed, in this case, shown in Fig. 2. Two wires were point-welded at both extremities of the specimen to apply the electric current. Two other wires were point-welded at the specimen central position, at 10 mm apart one from the other, to allow the electric voltage measure, as can be seen in the Fig. 2. Measurements were made in direct and inverse sense of application of current, thus eliminating the parasites currents. Therefore, the final voltage was the average of the two measured values. Continuous current of 30, 50, 80, 100 and 200 mA were used.

Scanning electron microscopy (SEM). The specimens used in the tensile test were examined in the scanning electron microscope Philips XL-30/MEF to check the type of fracture (intergranular or transgranular). The observation was made in the zone of fracture of the specimen after tensile test in the direction transverse to the direction of rolling.

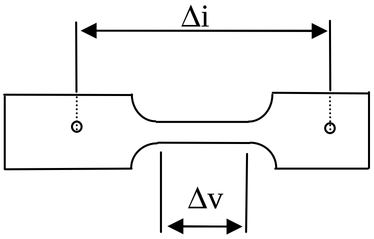


Figure 2. The four wire method employed in the measures of electrical resistivity

3. EXPERIMENTAL RESULTS AND DISCUSSIONS

Table 2 shows the values found for the yield stress and rupture stress, for stainless steel AISI 304 and AISI 348 L*. Each experimental value obtained represents the average of four specimen.

Table 2. Yield stress and rupture stress for stainless steel AISI 304 L and 348 L*

Material	σ_e(MPa)	σ_r(MPa)	ε(%)
304	164.11 ± 7.51	464.58 ± 21.21	68.12 ± 4.77
348 L*	190.72 ± 7.59	498.92 ± 19.75	52.71 ± 3.14

Table 3 shows the values of microhardness of stainless steel AISI 304 and AISI 348 L*. Each value represents the average obtained from four specimen, and, in each specimen were performed five measures.

Table 3. Microhardness values for stainless steel AISI 304 and 348 L*.

Material	Microhardness ($HV_{0.1}$)
304	189.7 ± 2.4
348 L*	199.3 ± 2.2

As seen in table 2, the values of yield stress and rupture stress for steel AISI 348 L* are higher than those of AISI 304. This indicates a higher ductility of the steel AISI 304, confirmed also by the values of microhardness obtained (table 3), which is lower for the steel AISI 304.

The increase in microhardness was also observed in AISI 321 austenitic stainless steel due to the addition of small amounts of Nb (0.1% by weight) [5].

Table 4 shows the values of electrical resistivity of stainless steel AISI 304 and AISI 348 L*. Each value represents the average obtained from four specimens, and, in each specimen five measures were performed. These values are consistent with those ones cited [6]: 80.2 $\mu\Omega$cm at 28.0 ºC for the steel AISI 304 and 75.5 $\mu\Omega$cm at 26.2 ºC for the steel AISI 347. Although the steel AISI 347 is not the object of study of this work, its chemical composition is similar to the steel AISI 348 L* (with N). It was used for a comparison scope once the values for the AISI 348 L* was not available.

Table 4. Values of Electrical Resistivity for stainless steel AISI 304 and 348 L*

Material	$\rho(\mu\Omega\text{cm})$
304	85.82 ± 8.02
348 L*	79.53 ± 7.43

Figs. 3 and 4 show the morphology of the fracture zone for stainless steel AISI 304 and AISI 348 L* respectively.

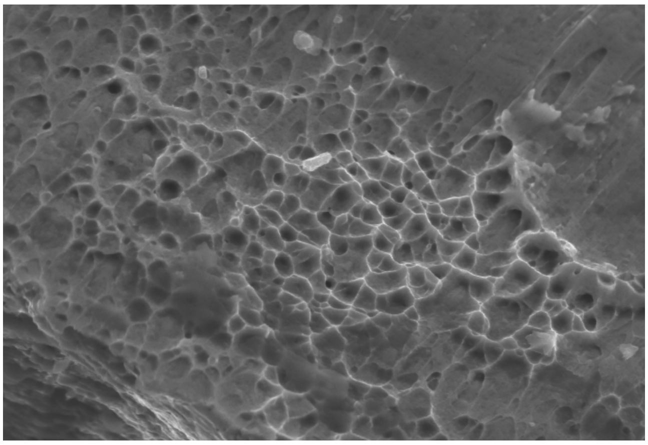


Figure 3. Appearance of fracture morphology steel AISI 304, in the cross direction rolling. 2000 X.

Note the presence of dimples on both steels, showing a transgranular fracture, which, is desirable behavior from the viewpoint of structural material in reactors. The largest amount of dimples observed in steel AISI 348 L* (Fig. 4) is probably due to the effect of N, which acts as the grain size refiner.

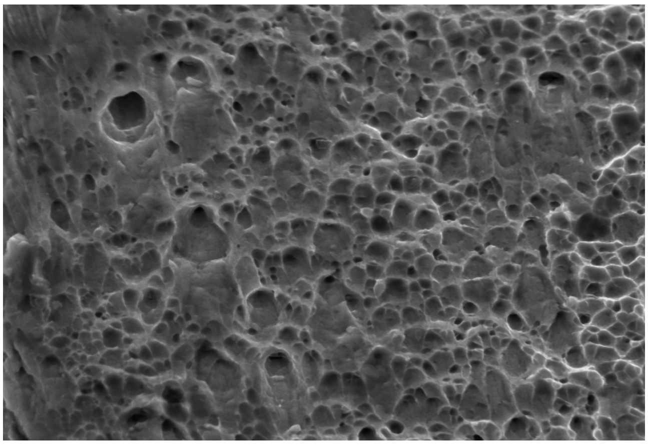


Figure 4. Appearance of fracture morphology steel AISI 348 L*, in the cross direction rolling. 2000 X.

Figs. 5 and 6 show the microstructures obtained by transmission electron microscopy for the steel AISI 304. There is a reasonable amount of dislocation, mainly near the grain contour. In Fig. 5, the dislocation are aligned in several groups.

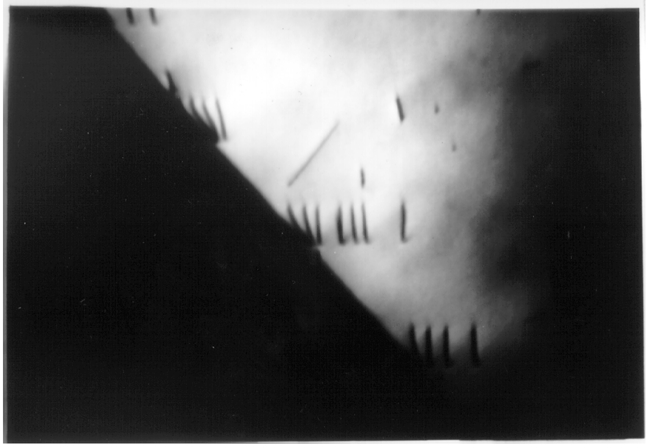


Figure 5. Microstructure steel AISI 304, in the cross direction rolling: 13400 X

Figs. 7 and 8 show the microstructures obtained by transmission electron microscopy for the steel AISI 348 L*. There is a number of dislocation similar to AISI 304 steel, but they are not aligned.

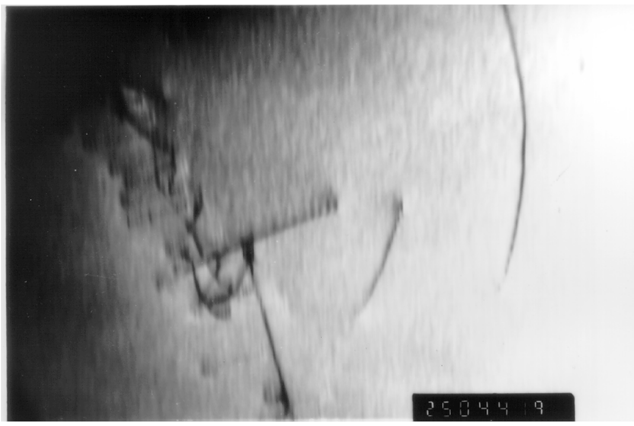


Figure 6. Microstructure Steel AISI 304, in the cross direction rolling: 20700 X

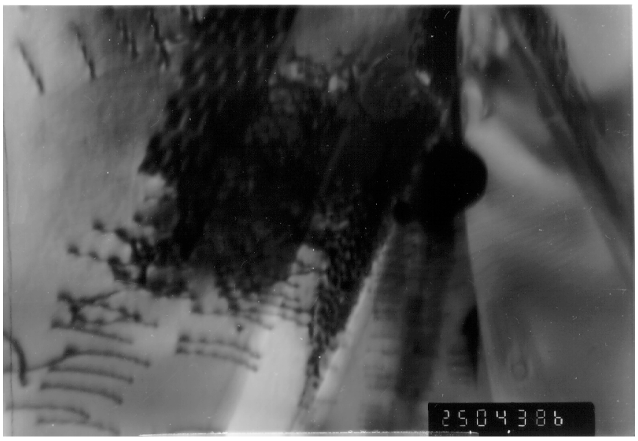


Figure 7. Microstructure Steel AISI 348 L*, in the cross direction rolling. 20700 X

Figure 8. Microstructure Steel AISI 348 L*, in the cross direction rolling. 20700 X

In order to continue this research, the next step is to complete the design and construction of the CIMAT irradiation device, which will allow the irradiation of the referred samples in the IEA-R1 research reactor. After the samples irradiation, the properties of steel AISI 304 and AISI 348 L* will be compared under the conditions pre and post-irradiated.

4. CONCLUSIONS

This work showed the characterization of mechanical and electric properties such as yield and rupture stress, microhardness and resistivity of the steel AISI 304 and AISI 348 L*. It was also analyzed the zone of fracture of specimen after the tensile test in the direction transverse to the rolling by means of scanning electron microscopy.

After the tensile test, the microstructure was observed in the steel AISI 304 and AISI 348 L* in the direction transverse to rolling through transmission electron microscopy.

The mechanical properties (yield stress and rupture stress and microhardness) of the steel AISI 348 L* are higher than in the steel AISI 304, while its elongation is lower. These facts show a higher ductility for the steel AISI 304. The electrical resistivity is consistent with the literature for steels AISI 304 and AISI 347**.**

The steel AISI 348 L* instead of the steel AISI 304, is desirable for use in reactor structural components due its low C, that promotes a decrease of the $Cr_{23}C_6$ precipitates in the grain boundaries, inhibiting the intergranular corrosion. On the other hand, the mechanical strength was improved due to the addition of Nb and N.

In a second phase of this work, we intend to characterize the properties of steel AISI 304 and 348 L* in the post-irradiated condition and compared with those ones in the pre-irradiated condition.

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