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The Occurrence of Laves Phase in Fe-15%Cr-15%Ni Austenitic Stainless Steels Containing Niobium

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Abstract

The Fe-15%Cr-15%Ni austenitic stainless steel has a fully austenitic microstructure, which is free of δ-ferrite and intermetallic phases. The formation of strain-induced martensite is also not observed. The effect of niobium additions in Fe-15%Cr-15%Ni stainless steel microstructure was studied. Additions of 0.5, 1.0 and 2.0 wt.% of niobium were done in the stainless steel studied. The as cast microstructure of the studied steels were characterized and analyzed by using several techniques. In the as cast steels samples, the results show absence of magnetism. The results also show the niobium addition caused an enlargement of the solidification interval and propitiates Laves phase formation. The Laves phase is hard and fragile and its formation between austenite dendrites causes loss of toughness. The mechanical test done made it possible to observe crack propagation alongside the fragile Laves phase.

Keywords: Stainless steel, Laves Phase, Mechanical Properties, Solidification.

Introduction

The Fe-15%Cr-15%Ni austenitic stainless steel has a fully austenitic microstructure, which is free of δ-ferrite and intermetallic phases such as sigma and chi phases because to their low Cr/Ni ratio and absence of molybdenum (1,2). The formation of the deformation or strain-induced martensite is also not observed in this kind of austenitic stainless steel (1,2). The effects of niobium addition in stainless steels have been widely studied. Niobium reacts with carbon resulting the NbC and thereby hinders the precipitation of $M_{23}C_6$, which reduces the susceptibility of the steels to intergranular corrosion (3).

The objective of this work is study the effect of niobium additions in Fe-15%Cr-15%Ni austenitic stainless steel microstructure. Nominal additions of 0.5, 1.0 and 2.0 wt.% of niobium were done in the stainless steel studied. The carbon in the alloys studied was maintained below 0.03% to minimize the NbC precipitation and the residual elements like P, S, Cu, Sn and As were also kept low. The microstructure and mechanical properties of the as cast alloys were analyzed by using several complementary techniques as X-ray diffraction, optical and scanning electron microscopy with dispersive energy analysis, and Vickers hardness. The presence of magnetic phases was determined with a ferritoscope. Tension tests were also done to evaluate mechanical properties.

Materials and Methods

The alloys with different niobium contents were prepared by vacuum induction melting from high purity starting materials. The chemical compositions of the steels studied are shown in table 1.

The microstructures of the as cast alloys were analyzed by using optical and scanning electron microscopy with energy dispersive analysis (EDS). The metallographic sample preparation consisted of grinding with up 2400-grit paper, followed by 1 mm diamond polishing and further electrolytic polishing was also performed. The composition of the polishing electrolyte was 950 ml acetic acid and 50 ml perchloric acid. The electrolyte was kept at 13°C and the samples were polished at 80V for 80 seconds. After electrolytic polishing, the samples were etched with V2A-Beize at

70°C for 20 to 60 seconds. The compositions of the different phases were determined by energy dispersive analysis.

Table 01. The chemical compositions of the steels studied (in wt.%).

Element	N00	N05	N10	N20a	N20b
C	0.02	0.02	0.02	0.02	0.03
Si	0.59	0.48	0.53	0.57	0.41
Mn	0.53	0.43	0.52	0.47	0.51
P	0.006	0.006	0.006	0.006	0.006
S	0.013	0.012	0.012	0.012	0.008
Cu	0.02	0.04	0.01	0.01	0.02
Cr	14.4	14.7	15.1	14.6	13.4
Al	<0.005	<0.005	<0.005	0.005	0.013
Sn	0.002	0.002	0.001	0.001	0.001
As	0.002	0.001	0.002	0.002	0.004
Ni	15.0	15.1	14.8	14.8	15.2
N	0.0084	0.0081	0.0075	0.0081	0.028
Mo	0.01	0.01	<0.01	<0.01	<0.01
Nb	<0.002	0.44	0.89	1.74	1.89

The crystal structures of the different phases were determined: (a) by X-ray diffraction measurements on polished surfaces with the aid of a diffractometer; and (b) by analysis of Debye-Scherrer camera data residue extracted after dissolution of the matrix. In both cases, Mo Kα radiation was used. The presence of magnetic phases, if any, was determined by using the magnetic induction method (a ferritoscope with 0.1% detection limit).

Tension tests were done to determine mechanical properties as yield strength, tensile strength and percent elongation. Tension testing was realized by using round bars specimens with 5 mm diameter and 25 mm length of gauge length. A kind of mechanical test was also done to evaluate the toughness of the steels studied. The test consisted of compressing the metallographically prepared samples. *Figure 1 shows, schematically, the sample and* the stress applied on it. By using this test, crack propagation can be easily observed. Vickers hardness was also measured, by using load of 10 kgf, to compare mechanical properties of the steels studied.

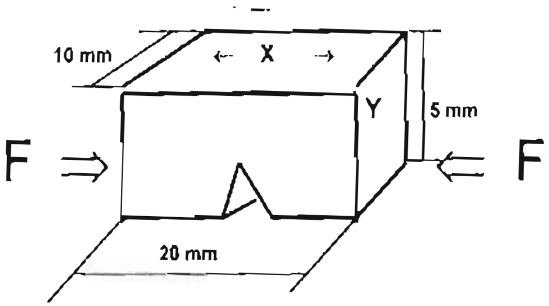

Figure 01. Schematic drawing of the sample for mechanical test. X is the surface metalographically prepared. Y is the surface where compression force (F) was applied.

Results

In the as cast steels samples, the results show absence of magnetism. On the other hand, an intermetallic phase was detected in the four steels containing niobium. The amount of the intermetallic phase is related to the niobium content. In the high niobium alloys (N10, N20a e N20b) the intermetallic phase formed a continuous network and the volumetric fraction of the intermetallic phase is proportional to niobium wt.%. Figures 2 and 3 show the microstructure of the steel containing about 2 wt.% of niobium.

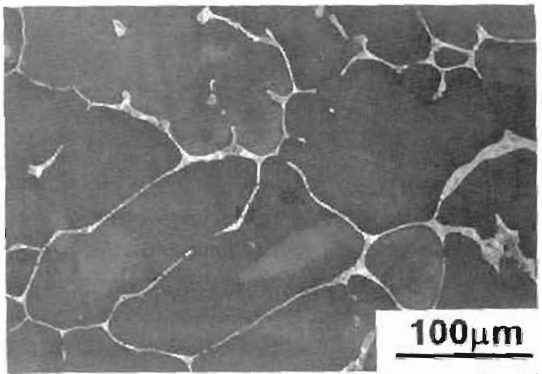

Figure 02. As cast sample containing about 2 wt.% of Niobium (N20a). Scanning electron microscopy. Etching: V2A-Beize.

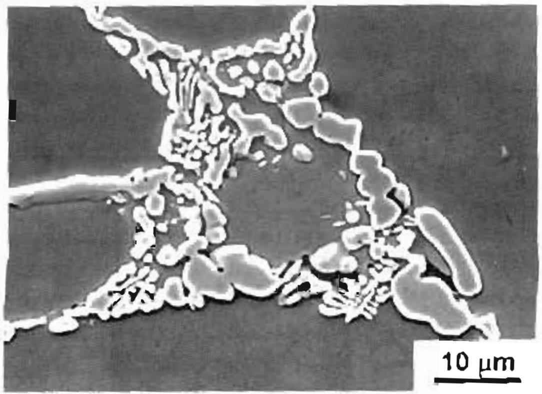

Figure 03. As cast sample containing about 2 wt.% of Niobium (N20a). Scanning electron microscopy. Etching: V2A-Beize. Detail of the micrograph of figure 2.

Energy dispersive analysis results shown high Fe and Nb contents and low Ni, Cr and Si contents, in the intermetallic phase. On the other hand, the results of the matrix analysis showed the absence of Nb. The intermetallic phase formed during solidification was identified as Laves phase by analyzing X-ray diffraction data. Table 2 shows the results of chemical composition and table 3 shows the results of the Laves phase lattice parameters in the steels studied. However, a more detailed examination of the microstructure revealed the presence of small quantities of NbC and mixed oxide particles. Figure

4 shows schematically the Fe-15%Cr-15%Ni-0.03%C-Nb phase diagram section proposed in this work.

Table 02. Chemical composition of the Laves phase in the studied steel determined by EDS analysis.

Element	at.%	wt.%
Fe	46.5 ± 0.9	41.6 ± 1.0
Ni	12.6 ± 0.5	11.9 ± 0.5
Cr	11.3 ± 0.3	9.4 ± 0.3
Nb	22.9 ± 0.7	34.9 ± 0.9
Si	6.8 ± 0.2	3.1 ± 0.1

Table 03. Lattice parameters of the Laves phase obtained by X-ray diffraction.

Phase	Lattice Parameters (nm)
$(Fe,Ni,Cr)_2(Nb,Si)$	a=0.476, c=0.786 c/a=1.651

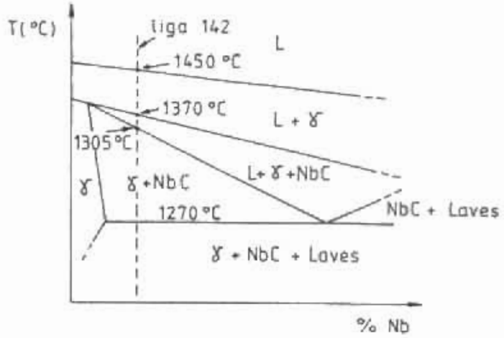

Figure 04. Fe-15%Cr-15%Ni-0.03%C-Nb phase diagram section. Schematic representation.

Niobium addition also caused an enlargement of the solidification interval. The effect of Niobium addition on *liquidus* and *solidus* lines of Fe-15%Cr-15%Ni system is shown in figure 5.

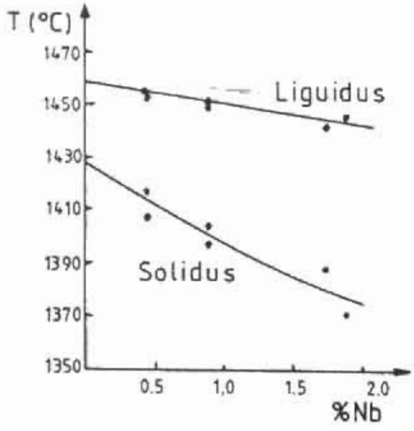

Figure 05. The effect of Niobium addition on *liquidus* and *solidus* lines of Fe-15%Cr-15%Ni system.

Figures 6, 7 and 8 show the results of the mechanical tests showed in figure 1. Cracks can be seen inside the regions containing the intermetallic phase. Mechanical tests were also done to determine the effect of niobium addition on mechanical properties of the studied steels. Figure 9 shows the mechanical properties with varying niobium contents in the studied steels.

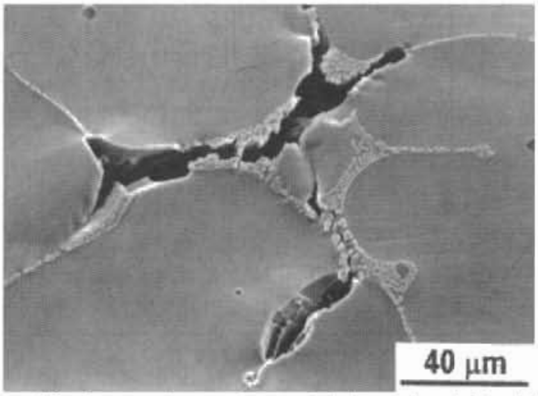

Figure 06. As cast sample containing about 2 wt.% of niobium (N20b). Scanning electron microscopy after mechanical test. Etching: V2A-Beize.

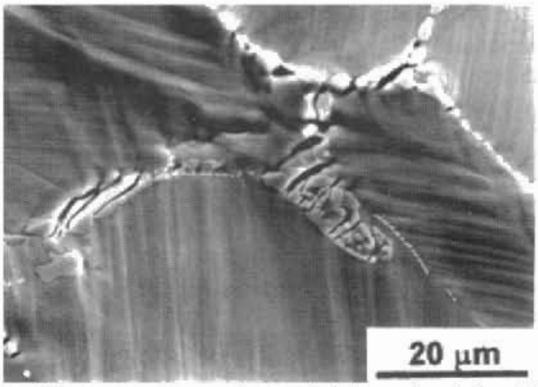

Figure 07. As cast sample containing about 2 wt.% of niobium (N20b). Scanning electron microscopy after mechanical test. Etching: V2A-Beize.

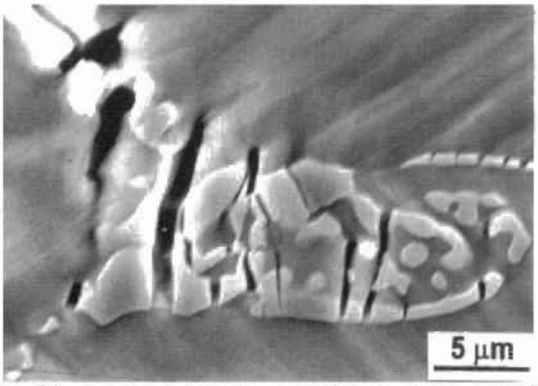

Figure 08. As cast sample containing about 2 wt.% of niobium (N20b). Scanning electron microscopy after mechanical test. Etching: V2A-Beize. Detail of the micrograph of figure 7.

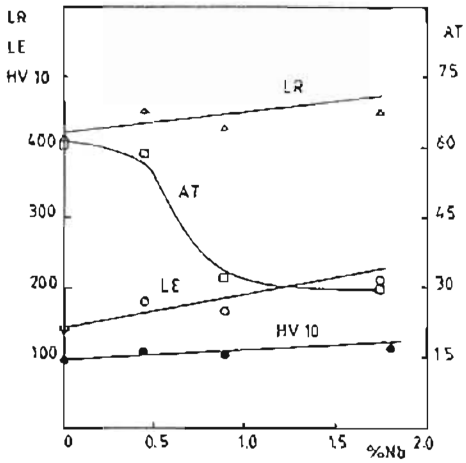

Figure 09. Vickers hardness (HV 10), yield point (LE, N/mm^2), tensile strength (LR, N/mm^2) and elongation (AT, %) variation with niobium contents in the studied steels.

Discussion

The results of magnetic measurements show absence of magnetism as predicted by using Schäffler diagram. Schäffler diagram is used to foresee stainless steels cast microstructures by using chromium and nickel equivalences. Figure 10 shows Schäffler diagram.

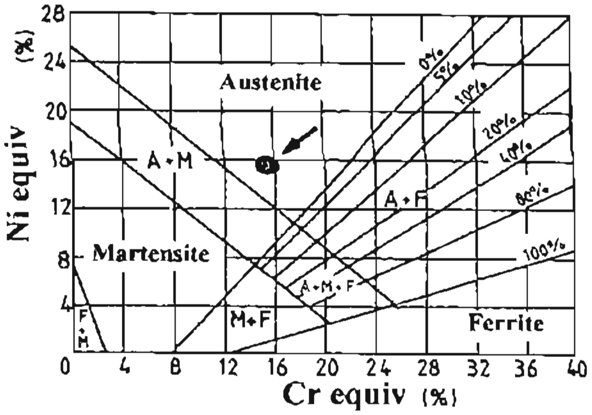

Figure 10. Schäffler diagram. The arrow indicates the alloys studied (4).
Cr equiv.=%Cr+1.4(%Mo)+0.5(%Nb)+1.5(%Si)+2(%Ti)
Ni equiv.=%Ni+30(%C)+0.5(%Mn)+30(%N)

The macrographic observation of the samples, after polishing and etching, showed a columnar zone near the surface and a more refined microstructure in the central region of the samples. Niobium additions caused a cast microstructure refinement.

Niobium addition also caused an enlargement of the solidification interval. This phenomenon certainly propitiated an increase in niobium segregation to solid-liquid interfaces and/or dendrite boundaries. Similar phenomenon occurs when molybdenum additions are done (5). After that, Niobium addition caused a lower in *liquidus* and *solidus* lines. *Liquidus* line lower is greater than *solidus* line lower.

Elements like molybdenum, titanium and niobium favor Laves phase formation. Niobium and silicon in the alloys studied containing niobium, are in combined form as a Laves phase of a type (Fe, Ni, Cr)$_2$(Nb, Si). The average electron concentration of the Laves phase in the niobium containing alloys was found to be 7.07. This value is very close to 7.0 obtained for the formula Fe$_2$Nb.

Laves phase formation consumed almost all niobium in the austenitic matrix. Laves phase forms coarse particles distributed alongside the dendrites. These coarse particles have a low hardener effect. Besides that the Laves phase network, rich in niobium, formed in or near dendrites causes a loss in ductile. Almost all niobium was concentrated in big particles.

The positive effect of niobium on hardness, yield point and strength is small as compared with negative effect on ductility (elongation). The large particles of Laves phase containing high levels of niobium have a small hardening effect. Using sample illustrated in figure 1, the hypothesis of nucleation and crack propagation into Laves phases was tested. The results show clearly the hypothesis was correct. Actually, the positive effect of niobium addition on hardness, yield point, strength is small if one compared the deleterious effect caused on toughness in the as cast materials studied.

Conclusions

In conclusion, the effect the niobium in the Fe-15%Cr-15%Ni austenitic stainless steel can be described as follows: 1) Niobium propitiates an enlargement of the solidification interval and the Laves phase formation. 2) The presence of the Laves phase had negative influence on the toughness of the steels studied. The Laves phase is hard and fragile and its formation between austenite dendrites causes loss of toughness. 3) The mechanical test done made it possible to observe crack propagation alongside the fragile Laves phase.

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