# EFFECT OF STABILIZATION ON CREEP RESISTANCE OF FERRITIC STAINLESS STEELS USED IN EXHAUST SYSTEMS

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**Abstract.** *The effect of ferritic stainless steels stabilization on creep resistance was studied. Steel dissolution technique for chemical extraction of precipitates and X Ray diffractometry were carried out in order to analyze the microstructure after creep tests. It was observed a beneficial effect of Nb on the creep resistance by Laves phase precipitation, mainly, in the grain boundaries. It was not observed any significant effect of titanium*

*Keywords*: Creep resistance, Ferritic Stainless Steels, Stabilization, Exhaust System.

## 1. INTRODUCTION

Vehicles exhaust system has been reaching a great evolution since last 20 years, because of environmental laws and high lifetime guarantee (Schimitt, 2002). Also, the development of high power cars takes to the use of materials with high temperature mechanical properties. These factors have been increasing the stainless steels use, mainly the ferritic stainless steels because of its lower cost than austenitic ones and higher competitiveness than aluminized carbon steels and cast iron (Schimitt, 2002; Fujita et al., 1996; Alves et al., 2002).

The aim of ferritic stainless steels stabilization is to remove C and N from solid solution through its precipitations with Ti, Nb, Va and others (Gordon and Bennekom, 1996; Oliveira, 2003). Ti is the most used stabilizing element, forming precipitates as TiN, TiC and Ti(C, N). Nb is less reactive and forms Nb(C, N) less stable precipitates, enough to prevent sensitization. Nb is efficient for grain size control, less oxidant and it does not have S and Cu affinity (Bucher, 2004; Mantel et al., 1990). Big Ti precipitates propitiate a toughness reduction while fine Nb precipitates decreases the ductility. The dual stabilization becomes possible to obtain an increase of both mechanical properties (Lee et al. 1999; Hunter and Eagar, 1980; Wasko and Grubb, 1991).

The absence of other phases in ferritic stainless steels and its type of crystalline reticulate favors a higher atomic mobility, allowing a faster grain growth and a lesser coalescence temperature. On the other hand, in presence of austenite and second phase particles [Ti(CN) or Nb(CN)], it occurs a grain growth retardation and increase of coalescence temperature (Ohashi et al., 1980; Plumtree and Gullberg, 1980; Courtnall and Pickering, 1976).

The creep resistance of austenitic stainless steel AISI 304 is significantly higher than ferritic ones because of its higher mechanical resistance at high temperature. Johnson (1981) has observed in its studies of creep at 870°C that niobium mono-stabilized steels have higher creep resistance than titanium monostabilized and dual-stabilized steels. The best creep resistance is obtained with austenitic stainless steels. Niobium mono-stabilized steels present a creep resistance related directly to free niobium amount in solid solution ($\Delta Nb$). Schimitt et al. (2005) have observed at SAG-TEST 100h at 950°C, that creep total strain has significantly decreased to $\Delta Nb$ values down to 0.40%.

Microstructure analysis shows that creep resistance improvement is directly related to formation of Laves phase during the creep test. Laves phase is a hexagonal intermetallic composition, usually of $(Fe, Cr)_2 (Mo, Ti, Nb)$ type. Its precipitation occurs at the grains bulk and boundaries, being in the globular particle shape. Its structure is compact hexagonal type (a = 0,482nm and c = 0,787nm). It can be formed after a huge exposition at high temperatures, occurring the precipitation at 600-900°C temperature range (Schimitt et al., 2005; Davis, 1994).

Ferritic stainless steels do not present a significant effect of carbon content on creep resistance. The main difference is with the existence of stabilization: titanium (AISI 409L) or not (AISI 410L). The creep resistance of AISI 409 grade is probably assured by precipitation of a fine phase of TiC that effectively suppresses the creep deformation (Yamanaka et al., 1983).

In single-phase steels, grain size has usually an important role on creep resistance at high temperatures. For the ferritic stainless steels case, Johnson (1981) and Kah (1975) have observed a little significant effect of grain size on the creep resistance improvement.

At the same temperature, low carbon steels present creep behavior inferior to all stainless steels. Austenitic stainless steels have better performance than ferritic, while dualstabilized grade presents better performance than no stabilized one, which has a very similar behavior than monostabilized one (Kah and Hultgren, 1975).

The purpose of this article is to study the effect of the ferritic stainless steels stabilization with Ti and Nb and their precipitation on creep tests at temperatures that characterizes the hot part of the vehicles exhaust system.

## 2. MATERIALS AND EXPERIMENTAL PROCEDURE

### 2.1 Chemical composition and metallographical analysis

The materials studied were ferritic stainless steels stabilized with Ti, Nb and Ti+Nb (dual stabilization) with a nominal thickness of 1.50mm. Titanium and niobium free in solid solution ($\Delta$Ti and $\Delta$Nb) were determined by the equations proposed by Sato and Tanoue (1995) and Fujimura and Tsuge (1999), which depend on the type of stabilization (mono or dual). The chemical composition is presented in Table 1. 16Cr4Nb steel presents a very small quantity of niobium free in solid solution ($\Delta$Nb) compared to the others ferritic stainless steels studied.

Table 1 - Chemical composition of stainless steels (% wt).

| Steel | Cr | C | N | Ti | Nb | $\Delta$Ti | $\Delta$Nb |
|---|---|---|---|---|---|---|---|
| 16Cr5Ti | 16.26 | 0.026 | 0.011 | 0.47 | 0.01 | 0.34 | --- |
| 16Cr4Nb | 16.23 | 0.019 | 0.021 | 0.01 | 0.36 | --- | 0.05 |
| 17Cr2Ti2Nb | 17.05 | 0.011 | 0.013 | 0.20 | 0.19 | 0.14 | 0.14 |
| 18Cr1Ti6Nb | 18.01 | 0.014 | 0.009 | 0.13 | 0.56 | 0.08 | 0.49 |

$\Delta$Ti = Ti – 3.42 N – 4 C                    Ti stabilized steels

$\Delta$Nb = Nb – 7.74 (C+N)                    Nb stabilized steels

$\Delta$Ti = Ti – 3,42 N – (0.30 x 4C)        Dual stabilized steels

$\Delta$Nb = Nb – (0.70 x 7.74C)               Dual stabilized steels

Metallographical analysis with Villela's reagent was carried out in the 3 planes of the samples (longitudinal and transversal to cold rolling). Microstructure recrystallization and grain size measurement were analysed.

### 2.2 SAG TEST Creep resistance Tests

SAG TEST is a kind of creep resistance test developed at the Centre de Recherche d'Isbergues CRI. This test consists to put a flat rectangular specimen fixed at 4 points of its extremities in a device. It must have enough space under the specimen for its deformation without touching some surface. Figure 1 shows a schematical drawing of this device.
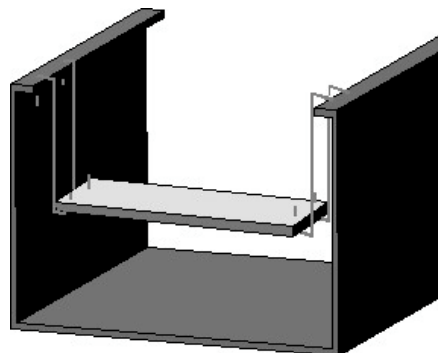


Figure 1 - SAG TEST device used on creep tests.

This device with the specimen is taken to a heat treatment oven and submitted to constant temperatures during pre-defined times. At a determined time interval, the specimens start to present a bending behavior. It is measured the height between the condition "zero" (before test and during the determined time interval, procedure known as "arrow measurement".

In this research SAG TEST specimen had 25mm width and 205mm length with length parallel to rolling direction. Two temperatures were considered on SAG TEST: 850 and 950ºC; the determined time interval were: 1, 25, 50 and 100h. It was tested two specimens for each temperature.

After the SAG TEST, the microstructures were characterized by grain size and oxide layer measurements on optical micrographs. Moreover, the precipitates were analyzed by metal dissolution technique on X ray diffractometer and SEM.

**2.3 - Analyses onX Ray diffractometer of precipitates obtained by dissolution technique**

The steel dissolution technique for chemical extraction of precipitates was carried out in creep specimens tested at 850 and 950ºC. Chemical dissolution technique consisted of sample chemical's attack with "Berzelius" reagent, followed by paper filtering and drying (Garzon et al., 2000). "Berzelius" reagent was prepared as presented in Table 2.

Table 2 - Chemical composition of reagent Berzelius.

| Quantity | Compound |
|----------|----------|
| 320g | dihidrated Cooper chloride ($CuCl_2 2H_2O$) |
| 280g | Potassium chloride (KCl) |
| 20g | Tartaric acid ($HOOC[CHOH]_2COOH$) |
| 1.85 l | Destilled water |
| 150ml | Chloride acid (HCl) |

The chemical dissolution was made at room temperature in 300ml Berzelius solution Becker with magnetic agitation. The samples were cut and washed in order to have around 2g weight. The chemical attack time was around 6 hours in order to have the complete dissolution of the steels. After that, the filtering stage of the dissolution product was carried out in a boron-silicate fiberglass filter. Once all the liquid filtering was completed, the filter was washed with current water. The drying of the filter with dissolved material was made during one hour in a warm oven at 100ºC. After that, SEM and X Ray diffractometry analyses were made.

The X Ray diffractometry technique was used to identify the precipitates types extracted by dissolution technique of steels. It was used an equipment X'PERT PROMPD Philips with goniometric Theta/Theta with copper anode tubes. The equipment's configuration was: incident beam with programmable divergence crevice, fixed mode in 0.5º, soller crevice of 0,04rad and 10mm mask. It was used the X'celerator detector of RTMS type at the diffracted beam - Real Teams Multiple Strip.

Data collect was carried out at 20-120º angle range (2 Theta). The phase's identification was obtained by comparison with pre-definitive standards using the database PDF-2 (Powder Diffraction File) edited and published by ICDD (International Center for Diffraction Data).

**3. Results and discussion**

**3.1 Microstructural analysis**

Microstructural cubes of the ferritic stainless steels are presented on Figure 3. The microstructure for all grades studied was homogeneous and constituted by recrystallized grains. The main difference found is the grain size, measured by the three concentric circles method, according to ASTM E112-95 Standard (1996), and presented on Figure 7.
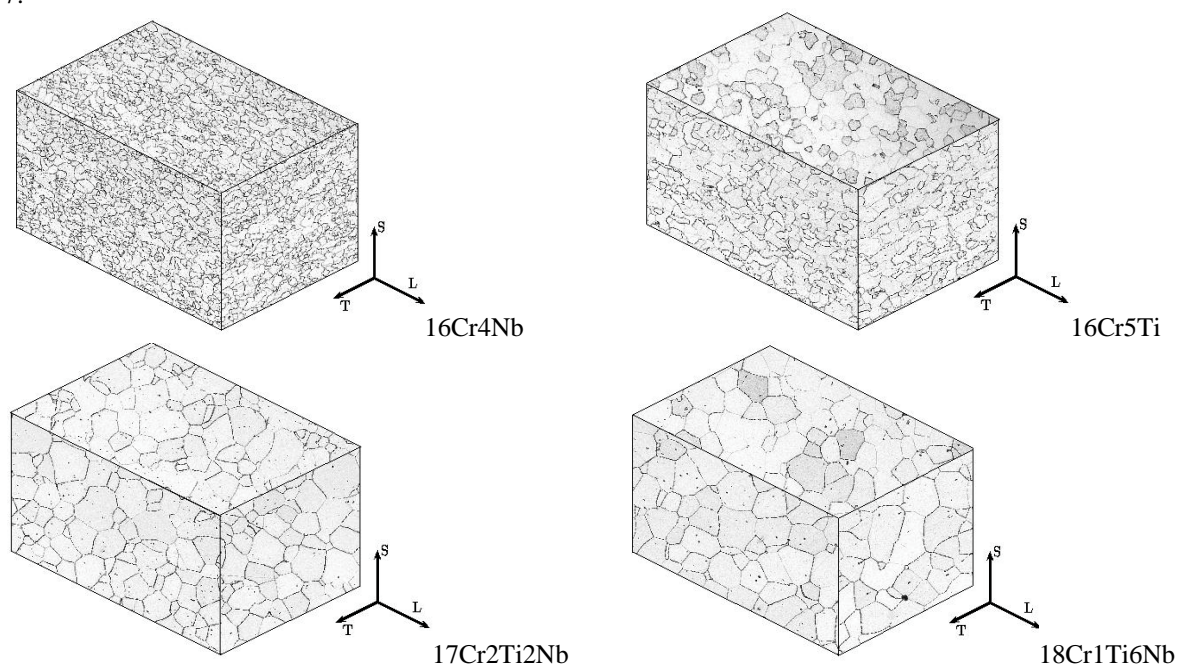


Figure 3 - Optical microstructural cube of the ferritic stainless steels, Villela's reagent. 200X.

The mono-stabilized steels have presented similar grain size, which are smaller than the dual-stabilized ones. This difference is more related to the steel fabrication standards condition, as heat treatment and, indirectly, to the chemical composition. The more niobium content, mainly ΔNb, increases the recrystallization temperature and so, the steel annealing must be done at higher temperatures and times, which allows the grain growth.

### 3.2 Creep tests

Figure 4 presents the results of the creep test "SAG TEST" at 850 and 950ºC. Higher the value of the arrow height, greater is the material deformation by its own weight and minor is its creep resistance.
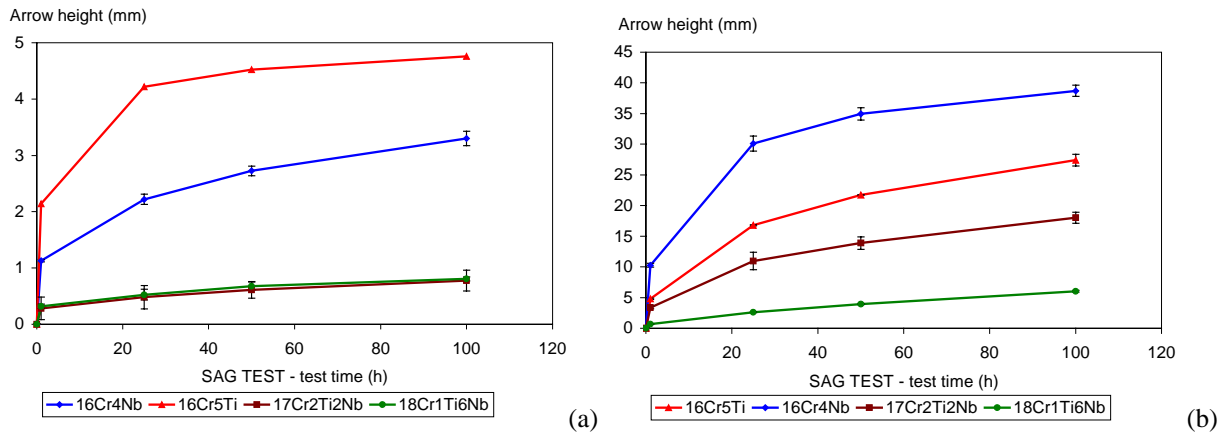
Figure 4 - Results of the creep test "SAG TEST". 850ºC (a) and 950ºC (b).

As observed on Figure 4, the ferritic stainless steels have presented a significant difference of creep resistance at the two test temperatures.

At 850ºC, the steels studied have presented a very small arrow height (always lesser than 5mm), that means, the steels have shown small deformation at this temperature. Dual-stabilized steels, with high free niobium content in solid solution (ΔNb), have presented a negligible alteration at the arrow height and an excellent creep resistance at this temperature. Mono-stabilized steels have presented a certain deformation.

At 950ºC, the steels studied have presented a significant deformation, mainly the mono-stabilized ones. So, the creep behavior of ferritic stainless steels has a direct relation to the temperature. Niobium mono-stabilized steel presents the worst creep resistance followed by titanium mono-stabilized steel. Dual-stabilized steel 18Cr1Ti6Nb presents a small increase at the arrow height in relation to the temperature of 850ºC, that means, the best creep behavior.

In terms of chemical composition, it was observed that titanium has not presented significant effect on the creep resistance of ferritic stainless steels. On the other hand, it was verified that the main effect of niobium was found as considering free niobium in solid solution (ΔNb) and not its total amount. Figure 5 presents the ΔNb effect on the arrow height measured at SAG TEST at 850 and 950ºC for 100 seconds.
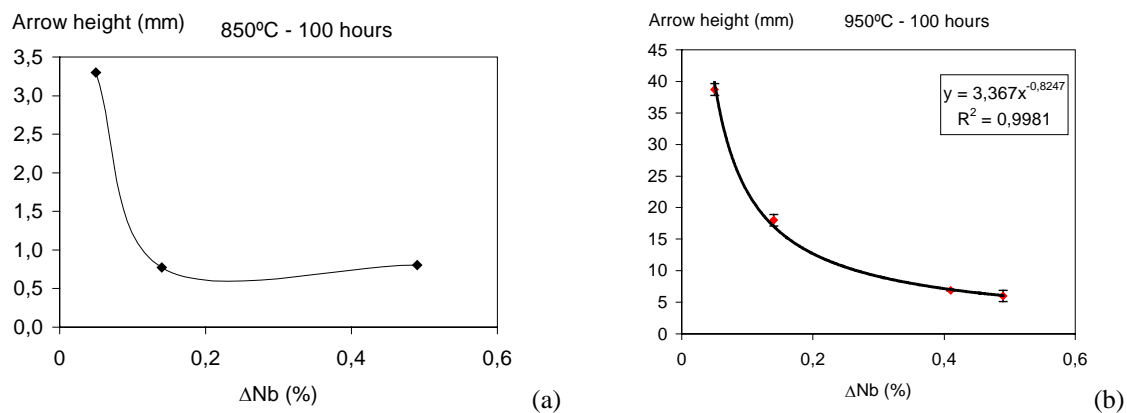
Figure 5 - Effect of ΔNb on the arrow height - SAG TEST (t = 100h) at the temperatures: (a) 850ºC and (b) 950ºC.

As verified on Figure 5, ΔNb has presented a significant effect on diminishing the arrow height, that means, increasing the creep resistance for both temperatures. At 850ºC, it was observed that creep resistance is significantly improved by a ΔNb content equals to 0.15% and that, up to this value, the arrow height becomes constant. At 950ºC, it

was possible to trace a tendency line and to get a mathematical equation relating ΔNb and the arrow height. In this case, the highest reduction of the arrow height was obtained to values equal to 0.35%. ΔNb. It was observed that the arrow height is constant up to this value.

Microstructural analysis was carried out at creep specimens in optical microscope. Figure 6 presents the results for both temperatures used. As observed, a microstructural alteration exists with the increase of the temperature in the creep test, which has justified the different steel behavior at these temperatures. This microstructural alteration can be explained by grain growth and precipitation/dissolution of niobium rich phases.
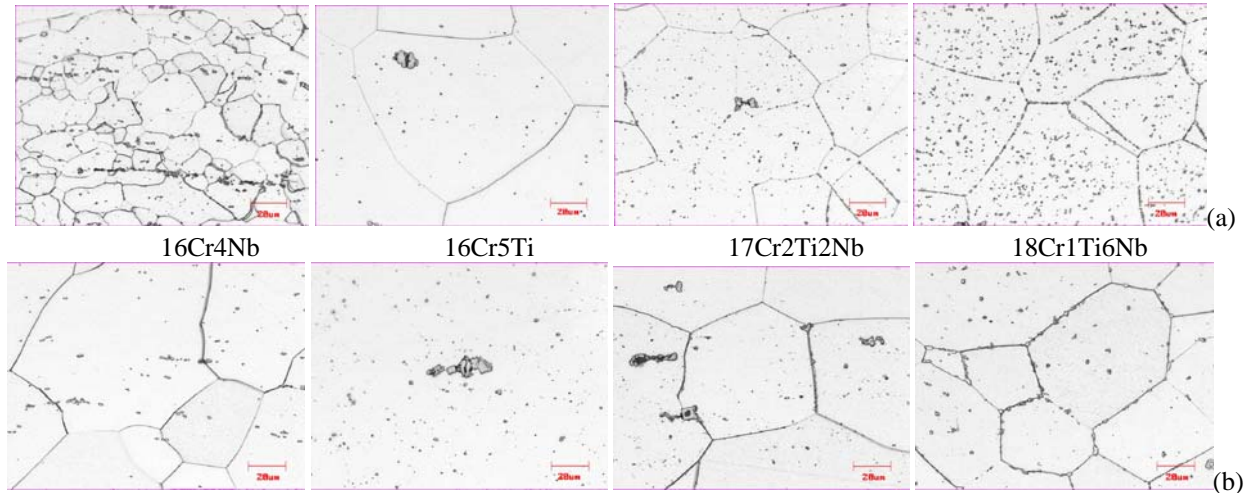


| 16Cr4Nb | 16Cr5Ti | 17Cr2Ti2Nb | 18Cr1Ti6Nb |

Figure 6 - Microstructural analysis of creep samples: (a) 850ºC (b) 950ºC.

Figure 7 presents the result of grain size measurement of the steels after creep tests at the two temperatures (100 hours) and makes a comparison with the initial grain size of the samples.
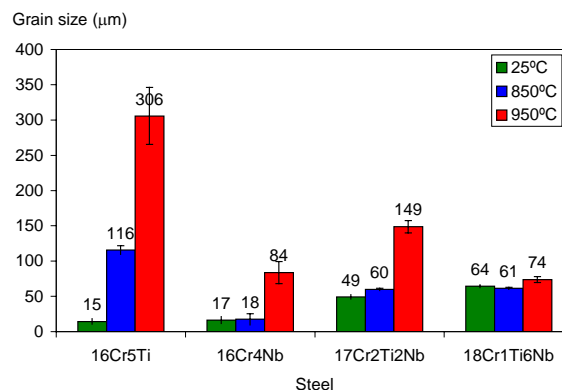


Figure 7- Evolution of the grain size during "SAG TEST" - time: 100h.

As observed on Figures 6 and 7, the evolution of grain size in function of the temperature is directly related to the chemical composition of ferritic stainless steel. Moreover, it is verified that the main alteration of grain size was obtained at 950ºC in relation to the condition before the test, except for the titanium mono-stabilized steel (16Cr5Ti).

At 850ºC, niobium stabilized steels have not presented significant variation at the grain size in relation to the initial condition, while titanium mono-stabilized steel has shown a significant increase. As verified on Figure 6 the niobium stabilized steels present precipitation of niobium rich phases in the bulk and grain boundaries. In the case of grain boundaries, these precipitates act as pinning and inhibit the grain growth, as discussed by Schimitt (2002) and Mantel (1990).

As verified on Figure 6, the microstructure of steels studied at 950ºC has presented a lesser amount of niobium precipitates in the bulk as well as in the grain boundaries, indicating that they have been dissolved at this temperature. Sawatani et al. (1982) have already observed the dissolution of niobium precipitates in this temperature. in a technique of residue extracted by electrolysis. So, the grain growth for niobium stabilized steels was observed, excepted to 18Cr1Ti6Nb. The titanium mono-stabilized steel has continued to present grain growth, superior to the others steels.

Dual-stabilized steel 18Cr1Ti6Nb, as shown on Figure 6, has presented precipitation in the bulk as well as, mainly, in the grain boundaries. As discussed previously, this precipitation in the grain boundaries hindered the grain growth by the pinning effect.

The dissolution technique of steels was carried out for chemical extraction of precipitates from SAG TEST specimens. The aim was to identify the precipitate types found in each temperature and analyzes them in SEM and X-ray difractometer. Figure 8 presents SEM analysis of the precipitates obtained by dissolution technique.
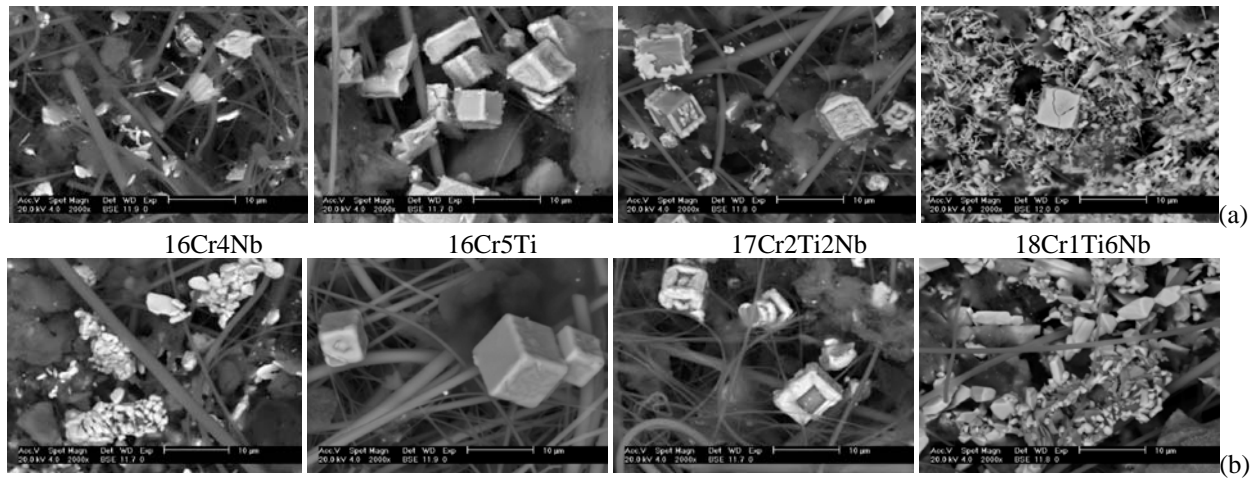


| 16Cr4Nb | 16Cr5Ti | 17Cr2Ti2Nb | 18Cr1Ti6Nb |

Figure 8- Analysis in SEM of the precipitates obtained by the steel dissolution in creep samples. (a) 850ºC; (b) 950ºC.

Through the dissolution technique of the steels followed by filtering and drying, it was possible to analyze the morphology and to define very well the precipitates type. The results obtained with this technique were coherent with the optical microscope analysis.

Table 3 presents the identification of the precipitates type observed in the creep specimens at 850 and 950ºC. The phases identification was carried out in X-ray difractometer using the PDF-2 database.

Table 3 - Identification of the precipitates observed by X-ray difractometry.

| Steel | 850ºC | 950ºC |
|---|---|---|
| 16Cr4Nb | Nb (C, N) | Nb (C, N) |
| 16Cr5Ti | TiN | TiN |
| | $Al_2O$ (*) | $Al_2O$ (*) |
| | TiC | TiC |
| 17Cr2Ti2Nb | $Fe_2Ti$ | Nb (C, N) |
| | Nb (C, N) | TiN |
| | TiN | TiO |
| | TiO | |
| 18Cr1Ti6Nb | TiN | TiN |
| | Nb (C, N) | Nb (C, N) |
| | (Nb, Ti) C | (Nb, Ti) C |
| | $Fe_2Ti$ | $Fe_2Ti$ |
| | $Fe_2Nb$ | $Fe_5Nb_3$ (*) |

(*) Possible presence.

The precipitates have presented two types of phase structure:
Cubical of centered face CFC: Nb (C, N); TiN; $Al_2O$; TiC; (Nb, Ti) C.
Hexagonal: TiO; $Fe_2Ti$; $Fe_2Nb$ and $Fe_5Nb_3$.

Niobium monostabilized steel (16Cr4Nb) has presented only Nb (C,N) type precipitate for both temperatures. The precipitates shape is flat, positioned separately or together. The Nb(C, N) precipitates coalescence was observed at 950ºC. It is important to observe that NbC and NbN precipitates present a same crystalline arrangement, which makes no possibility for differentiation in X-ray difractometer. So, it was defined to represent these precipitates as Nb(C, N) type.

Titanium mono-stabilized steel has presented TiN and TiC precipitates. TiN precipitate presents a cube shape while TiC one is found on the extremities of the cube diagonal axe. The possible presence of $Al_2O$ micro-inclusions was also observed. It was not verified alteration at type and morphology of precipitates between the two test temperatures.

Dual-stabilized steel 17Cr2Ti2Nb with intermediate titanium and niobium contents has presented TiN precipitates, but without TiC presence on the extremities. Moreover, the presence of TiO micro-inclusion was observed. The fine precipitates observed at mettalographical analysis ( Figure 6) were identified as "Laves phase" of $Fe_2Ti$ and $Fe_2Nb$ type at 850ºC. The Nb(C, N) precipitates have precipitated around TiN ones (with cubical shape). With the increase of temperature to 950ºC, the Laves phase dissolution and Nb(C, N) precipitates coalescence around the TiN precipitates have occurred.

Dual-stabilized steel 18Cr1Ti6Nb has also presented Nb(C, N) precipitates around TiN ones, which confirms this kind of precipitation for dual-stabilized steels. It was also observed the presence of (Nb, Ti)C precipitates at both temperatures. At 850ºC, it was verified again the presence of $Fe_2Ti$ and $Fe_2Nb$ Laves phase. As observed on Figure 8, the $Fe_2Nb$ Laves phase has presented the shape of small needles and corresponds to the precipitates observed in the grains bulk and boundaries at the optical microscope analysis.

At 950ºC, it was observed the dissolution of $Fe_2Nb$ Laves phase while $Fe_2Ti$ type has continued to precipitate. The precipitation of iron and niobium rich phase was observed in the grain boundaries, whose identification by X-ray difractometer showed possible presence of $Fe_5Nb_3$. According to literature (2005) this kind of precipitates could be $Fe_2Nb_3$ type, but its identification was not possible due the absence of this file in our X-ray difractometer database. As observed on Figure 8(d), $Fe_5Nb_3$ precipitates have a circular shape and are presented continuous (jointed together).

Comparing Figures 8 to 6, it was observed that $Fe_5Nb_3$ has precipitated in the grain boundaries, keeping its shape after the ferritic matrix dissolution. As discussed previously and observed by Schimitt (2005), the precipitation of iron and niobium rich fine phase act as pinning of grain hindering its growth. Moreover, the presence of precipitates in the grain boundaries hinders the grain free sliding, one of the creep mechanisms proposed by Honeycombe (1984). So, the highest presence of free niobium in solid solution allows the precipitation of iron and niobium rich phases in the grain boundaries and improves the ferritic stainless steels creep resistance.

Similar to observed by Kah and Hultgren (1975), it was verified that ferritic stainless steel creep resistance is directly related to chemical composition, while grain size has a little significant effect on its improvement.

## 4. CONCLUSIONS

The main conclusions of this article are:

Ferritic stainless steels creep resistance is related directly to the niobium content free in solid solution (ΔNb). At 850ºC, the steels have presented a very similar creep resistance, observed by the small specimen deformation (arrow height). The increase of temperature to 950ºC has promoted the reduction of creep resistance and significant differences among the steels have been observed.

It was verified that the increase of free niobium (ΔNb) content up to 0,15% (850ºC) and 0.35% (950ºC) have promoted a significant improvement of creep resistance. After this value, the creep resistance started to have a constant behavior that means it does not have no alteration.

Through the precipitates extraction analysis, it was possible to identify the phases presented at each studied steel. Dual-stabilized steels, because of higher ΔNb contents, have shown the intermetallic phases precipitation of "Laves phase" type in the bulk and, mainly, the grain boundaries at 850ºC. The increase of temperature to 950ºC promoted the dilution of this phase and, for 18Cr1Ti6Nb steel, it was observed the precipitation of another phase ($Fe_5Nb_3$).

The precipitation of intermetallic phases in the grain boundaries promoted the improvement of creep resistance for dual-stabilized ferritic stainless steels, mainly. These precipitates have acted as pinning of grain boundaries hindering its growth as well as its movement. A direct effect of grain size in the creep resistance of steels studied was not verified.

## 5. ACKNOWLEDGEMENTS

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