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Fe_3Nb_3N precipitates of the Fe_3W_3C type in Nb stabilized ferritic stainless steel

A. Malfliet[a,*], W. Van den Broek[b], F. Chassagne[c], J.-D. Mithieux[c], B. Blanpain[a], P. Wollants[a]

[a] *Katholieke Universiteit Leuven, Department of Metallurgy and Materials Engineering, Kasteelpark Arenberg 44 – Box 2450, 3001 Heverlee, Belgium*
[b] *University of Antwerp, EMAT, Groenenborgerlaan 171, 2020 Antwerp, Belgium*
[c] *ArcelorMittal Isbergues Research Centre, Rue Roger Salengro, BP15, 62330 Isbergues, France*

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ABSTRACT

A Nb stabilized ferritic stainless steel with 0.45 wt.%Nb, 82 ppm C and 170 ppm N is investigated to reveal the nature of the precipitates present at 950 °C. In particular, Fe_3Nb_3X precipitates of the Fe_3W_3C type are analyzed with WDS and EELS to determine the light elements X stabilizing this phase in the steel. According to WDS on large precipitates after 500 h at 950 °C, the Fe_3Nb_3X phase contains 10.4 at.% N, 1.2 at.% O and 1.0 at.% C. Auger Electron Spectroscopy on the same precipitates confirms the presence of N. In addition, it is revealed that the C and O peaks observed with WDS result from surface contamination as they disappear after Ar sputtering. The presence of a N peak in the EELS spectra of small Fe_3Nb_3X precipitates which have formed after 6 min at 950 °C indicate that N stabilizes this phase already from the initial precipitation stage. With this analysis it is demonstrated that N is an effective stabilizer of Fe_3Nb_3X precipitates in ferritic stainless steels. The formation of this phase should therefore be considered when predicting the precipitation behavior of Nb in industrial Nb stabilized ferritic stainless steels containing residual N.

1. Introduction

The steel market for automotive exhaust systems has been characterized the last decade by an increased use of ferritic stainless steel at the expense of the austenitic grades. Comparing in general ferritic with austenitic grades, ferritic stainless steel grades have inferior mechanical properties at high temperatures, but are not subject to the highly fluctuating Ni prices. The aim for ferritic grades with comparable mechanical properties as the austenitic grades, has resulted among others in the development of Nb stabilized ferritic stainless steel grades. Nb alloying is reported to improve the mechanical properties of the steel through solid solution and/or precipitation strengthening [1–5]. In general, in a Fe–Cr–Nb–C–N steel, three kinds of precipitates can be observed, i.e. Fe_2Nb, $Nb_x(C,N)$ and a phase of the Fe_3W_3C type. According to Sim's work [4], Nb(C,N) and Fe_2Nb precipitates coexist in an industrial Fe–15Cr–0.38Nb–0.01C steel after 2 h heat treatment at 700 °C. After 1000 h Fe_3Nb_3C precipitates are observed. Fujita et al. [6] determined the solubility product of the Fe_3Nb_3C phase in different Fe–Cr–Nb–C–N ferritic stainless steel grades between 700 and 1000 °C. Their results indicate that the amount of Fe_3Nb_3C increases with ageing time and temperature and that Cr enhances the formation of this Fe_3Nb_3C phase. Chassagne et al. [5] observed precipitates with similar crystallography and stoichiometry as the Fe_3Nb_3C phase in an industrial Nb stabilized ferritic stainless steel, but as there was no clear presence of light elements in these precipitates they were referred to as Fe_2Nb_3 precipitates. It should be noted that in the previously mentioned studies dealing with the precipitation of Nb in ferritic stainless steel, there is no qualitative study of the concentration of light elements in the Fe_3Nb_3C phase. In compounds of the filled Ti_2Ni type to which the Fe_3Nb_3C phase belongs, different light elements in different concentrations can stabilize the same metallic compound. For example both Fe_3Nb_3N and Fe_6Nb_6O are known to exist [7,8]. It is therefore not excluded that in a Fe–Cr–Nb–C–N ferritic stainless steel N could as well be present in these assumed Fe_3Nb_3C precipitates. The general formula Fe_3Nb_3X will be used in this study to refer to this phase of the Fe_3W_3C type without specification of the element X stabilizing this phase.

This work aims to clarify the nature of the Fe_3Nb_3X precipitates in an industrial Fe–Cr–Nb–C–N steel. The same ferritic stainless steel as that in the work of Chassagne et al. [5] is selected.

2. Material and methods

Table 1 shows the chemical composition of the ferritic stainless steel used in this investigation. This steel has been subjected to the classical production process

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* Corresponding author. Tel.: +32 16 321279; fax: +32 16 321991.
E-mail addresses: annelies.malfliet@mtm.kuleuven.be (A. Malfliet), wouter.vandenbroek@ua.ac.be (W. Van den Broek), Francis.chassagne@arcelormittal.com (F. Chassagne), Jeandenis.mithieux@arcelormittal.com (J.-D. Mithieux), bart.blanpain@mtm.kuleuven.be (B. Blanpain), patrick.wollants@mtm.kuleuven.be (P. Wollants).

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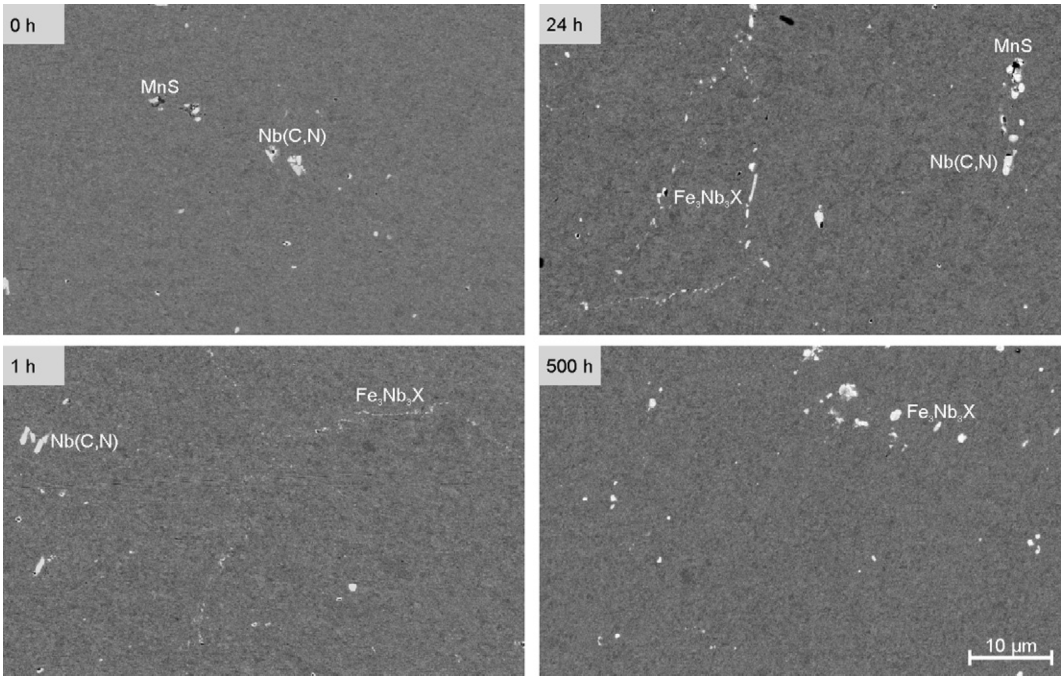

Fig. 1. BSE images of the evolution of the number and size of the precipitates with heat treatment time at 950 °C in the Nb stabilized ferritic stainless steel. The initial microstructure contains only $Nb_x(C,N)_y$ and MnS precipitates. During heat treatment small Fe_3Nb_3X precipitates form at the grain boundaries. The volume fraction increases with time until the equilibrium fraction is reached. The number of Fe_3Nb_3X precipitates initially increases. Thereafter, when precipitate coarsening takes place, it decreases.

for sheet, i.e. casting, hot and cold rolling to the desired thickness and annealing at 1050 °C for recrystallization. The samples are heat treated at 950 °C for up to 24 h in air. One sample is first sealed in an evacuated quartz tube to avoid excessive oxidation during a 500 h heat treatment at 950 °C. After heat treatment the samples are quenched in water. Samples for SEM are prepared by grinding, polishing and electrolytic etching in oxalic acid. SEM is used to visualize the location and distribution of the precipitates and to perform Energy Dispersive Spectroscopy (EDS) analysis on large precipitates. The volume fraction of precipitates is determined with the systematic point count method using image analysis. For each sample, five images with an area of 188 μm × 122 μm are analyzed. These images contain 2136 × 1263 pixels with a pixel resolution of 88 nm × 97 nm. The images are binarized and the amount of black and white pixels is counted to determine the area fraction of respectively the precipitates and the matrix. This area fraction is set equal to the volume fraction. The standard deviation is calculated to estimate the error.

Further characterization, especially of the small precipitates, is performed with TEM by using carbon extraction replicas and ion thinned specimens. The first step to prepare carbon extraction replicas is etching a polished sample for 4 min in a picric solution (20 g/l picric acid, 50 ml/l HCl and ethanol) followed by deposition of a carbon nanolayer. This layer, containing the precipitates, is removed from the sample by dissolving the steel matrix in the same picric solution. The carbon replica is positioned on a copper grid before inserting in the TEM. For the ion thinned films, the sample is first mechanically grinded and polished to a thickness of about 30 μm. With a Gatan ion thinner the sample is further thinned in the center with an Ar ion beam under an angle of first 15° and then 10°. Before inserting in the TEM, the sample is cleaned with a plasma cleaner. A Philips CM200FEG operated at 200 kV is used for Bright Field (BF) images, EDS and Selected Area Electron Diffraction (SAED) on carbon extraction replicas. EDS is mainly used to identify the precipitates, prior to determining their crystal structure with SAED. A qualitative Electron Energy Loss Spectroscopy (EELS) analysis is performed on a Philips CM30FEG operated at 300 kV in diffraction mode, and equipped with a post-column Gatan Imaging Filter.

Quantitative Wavelength Dispersive Spectroscopy (WDS) elemental analysis of the sample heat treated at 950 °C for 500 h is obtained from a JEOL JXA-8500F. The microprobe is operated at 10 kV using a probe current of 10 nA. For large precipitates (several μm) a probe size of 1 μm is used, while for smaller precipitates a spot is used. The intensity of the outgoing beam per unit wavelength is measured by a LDE2H crystal for C, a LDE1H crystal for O and N, a TAP crystal for Si, a LiFH crystal for Fe and Cr and a PETH crystal for Nb. Cr_7C_3, AlN and Al_2O_3 are chosen as standards for respectively C, N and O. To quantify Si, Cr, Fe and Nb, the pure elements are chosen as standards. ZAF is used for matrix correction method.

Auger Electron Spectroscopy (AES) is performed on a VG350f Microlab Auger system. The beam energy and tilt angle were respectively 10 kV and 30°. For ion sputtering a 3 kV Ar beam with an emission current of 10 mA is used.

3. Results

3.1. SEM analysis

Fig. 1 shows the evolution of the microstructure with heat treatment time. Initially the ferritic stainless steel matrix contains mainly $Nb_x(C,N)_y$, MnS and SiO_2 (not present in Fig. 1) particles which have formed during the production process. $Nb_x(C,N)_y$ precipitates appear white in BSE images, MnS and SiO_2 precipitates are black. The volume fraction of $Nb_x(C,N)_y$ is estimated to be 0.25 ± 0.04%. After 1 h at 950 °C, the latter precipitates are still present, but at the grain boundaries small Fe_3Nb_3X precipitates have formed. Both type precipitates appear white in the BSE image. The volume fraction Fe_3Nb_3X precipitates initially increases with time. The number of Fe_3Nb_3X precipitates after 24 h of heat treatment is comparable as after 1 h, but their size has increased. After 500 h at 950 °C, coarsening of the Fe_3Nb_3X precipitates is observed as the size of the Fe_3Nb_3X precipitates has further increased, but their number decreased. 100 white precipitates were analyzed with EDS. 98% were identified to be Fe_3Nb_3X precipitates. The other 2% could be $Nb_x(C,N)_y$ precipitates, but as they were either too small or completely surrounded by Fe_3Nb_3X precipitates, their nature was not clear from EDS analysis. The total volume fraction of the white appearing precipitates is 0.83 ± 0.08%. No MnS precipitates have been observed, but SiO_2 is still present.

Table 1
Composition of the ferritic stainless steel used in this investigation. Alloying elements with a concentration less than 0.05 wt.% are not shown. The remaining is Fe.

Element	Cr	Si	Nb	Mn	Ni	C	N	O
wt.%	14.8	0.576	0.453	0.192	0.112	0.008	0.017	0.003

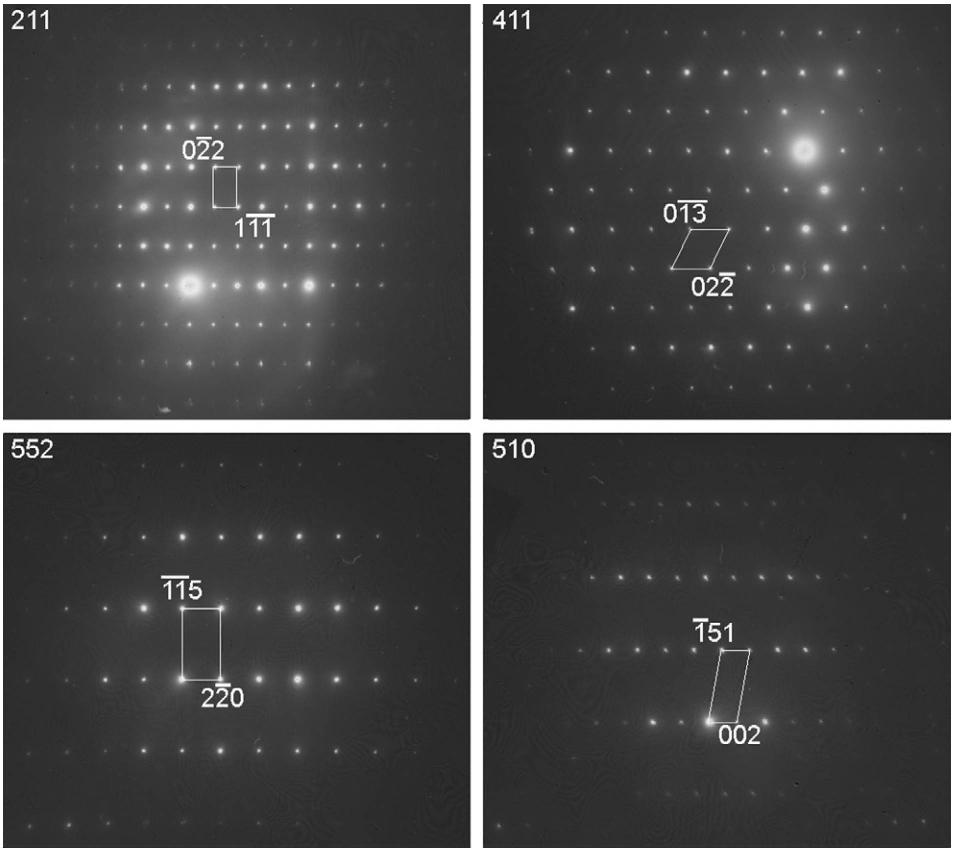

Fig. 2. Selective area electron diffraction patterns of the Fe_3Nb_3X precipitates by which the phase is identified as a face-centered cubic crystal with lattice parameter 1.1(3) nm.

3.2. *TEM analysis*

3.2.1. *SAED*

The crystal structure of the Fe_3Nb_3X phase is measured with SAED on TEM after extracting the precipitates on carbon replicas. The analysis of the diffraction patterns shown in Fig. 2 indicates that the Fe_3Nb_3X precipitates have a face-centered cubic crystal with lattice parameter 1.1(3) nm.

3.2.2. *EELS*

The Fe_3Nb_3X precipitates and the matrix of the Nb stabilized ferritic stainless steel heat treated for 6 min at 950 °C are analyzed by EELS on ion thinned TEM samples. EELS spectra which are representative for the matrix and the Fe_3Nb_3X precipitates in this sample are shown in Fig. 3. In the spectrum of the stainless steel matrix peaks on the position of Fe and Cr are observed as expected. Besides these, also a large peak of C and a smaller peak of O can be noticed. In the spectrum of the Fe_3Nb_3X precipitates peaks from Nb and N are observed which do not appear in the spectrum of the matrix. Comparing the two spectra, the Fe, Cr, Nb, N and O peaks are more pronounced in the spectrum of the Fe_3Nb_3X precipitates, but the C peak intensity has decreased with respect to the background.

3.3. *WDS analysis*

The concentration of light elements in the Fe_3Nb_3X phase is quantified with WDS measurements on large precipitates in a sample of the Nb stabilized stainless steel heat treated for 500 h at 950 °C. The WDS spectra and quantitative analysis for the elements Nb, Fe, Cr, Si, O, N and C in the Fe_3Nb_3X precipitates are given in Fig. 4 and Table 2. The main light element in the Fe_3Nb_3X precipitates is N. Besides C and O, it can be seen from Fig. 4 that also a small amount of P is present in the precipitates, but this could not be quantified.

3.4. *AES analysis*

From the WDS analysis of the Fe_3Nb_3X precipitates it follows that N is clearly present in these precipitates, but also a small amount of C and O is detected (Table 2). As it is known that C and O can easily come from surface contamination, the precipitates of the sample heat treated for 500 h at 950 °C are investigated with AES with Ar sputtering. Before Ar sputtering, the AES spectra of the (Fe,Cr) matrix and both the $Nb_x(C,N)_y$ and Fe_3Nb_3X precipitates show clear peaks of C and O. These are the grey spectra in Fig. 5. The spectra after 60 s of argon sputtering are shown in black in Fig. 5. In both the matrix and the precipitates the O peaks have significantly decreased. Due to the overlap between the O and the Cr peaks, the presence of a small amount of O cannot be excluded from the AES spectra of the (Fe,Cr) matrix and the Fe_3Nb_3X precipitates. The difference between the grey and black spectra nonetheless indicates that the main fraction of O measured without sputtering is from surface contamination. Peaks of both C and N peaks are distinguished in the spectra of the $Nb_x(C,N)_y$ precipitates. In the spectra of the Fe_3Nb_3X precipitates the N peak has become more distinct after Ar sputtering, indicating that this is the main light element in this phase.

4. Discussion

Before heat treatment of the Nb stabilized ferritic stainless steel at 950 °C, Nb is present in the $Nb_x(C,N)_y$ precipitates and in solid solution in the matrix. From the volume fraction of the $Nb_x(C,N)_y$ precipitates, the relative density of the matrix and the

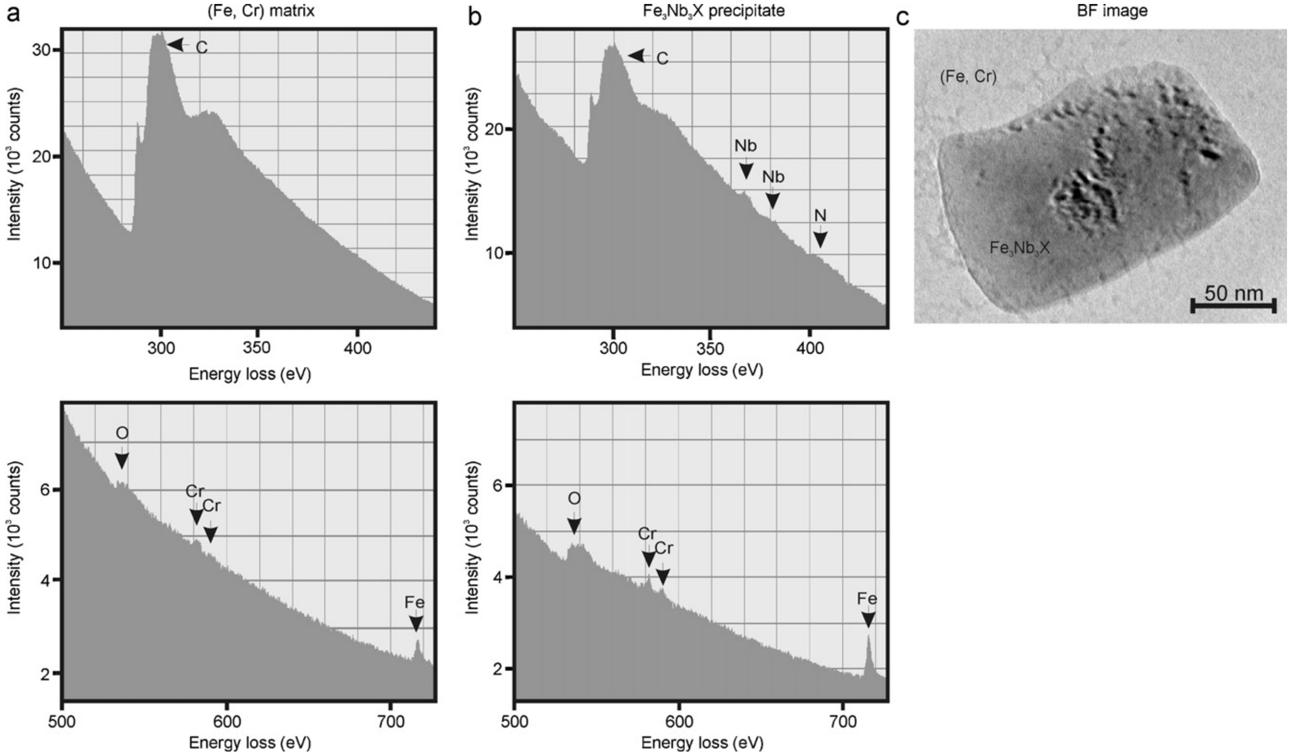

Fig. 3. EELS analysis of the light elements in the (Fe,Cr) matrix (a) and in small Fe_3Nb_3X precipitates (b). Both spectra show C and O peaks, but the N peak is only present in the Fe_3Nb_3X precipitates. (c) BF image of the matrix and Fe_3Nb_3X precipitates.

$Nb_x(C,N)_y$ phase and the concentration of Nb in the $Nb_x(C,N)_y$ phase, the amount of Nb precipitated can be calculated. Assuming that only the cubic Nb(C,N) phase is present with full occupancy of the (C,N) sites, about 0.24 ± 0.04 wt.% Nb has precipitated as Nb(C,N). The amount of C and N in this phase should then be 342 ± 53 ppm, but there is only 82 ppm C and 170 ppm N in the steel. Either the volume fraction is slightly overestimated or Nb is also present as Nb_2C or Nb_2N precipitates. Both the Nb(C,N) and $Nb_2(C,N)$ precipitates appear white in BSE images. Assuming that the volume fraction is overestimated and that all C and N have precipitated only as Nb(C,N), 0.18 wt.% Nb is present as Nb(C,N), thereby leaving 0.27 wt.% Nb in solid solution in the (Fe,Cr) matrix. If the volume fraction precipitates is not overestimated, the volume fraction of Nb(C,N)and $Nb_2(C,N)$ is calculated to be respectively 0.11% and 0.14% assuming that all C and N are equally divided between Nb(C,N) and $Nb_2(C,N)$. The amount of Nb precipitated is then 0.25 wt.%, leaving 0.20 wt.% Nb in solid solution in the (Fe,Cr) matrix. The amount of Nb in solid solution in the (Fe,Cr) matrix is therefore estimated to be between 0.20 and 0.27 wt.%.

During heat treatment at 950 °C the Fe_3Nb_3X phase precipitates at the expense of the $Nb_x(C,N)_y$ precipitates. Analogue to the calculation for the steel in the initial state, the amounts of Nb, C and N in the $Nb_x(C,N)_y$ and Fe_3Nb_3X precipitates after 500 h heat treatment can be calculated from their precipitated volume fraction. It is assumed that both C and N are present in the Nb(C,N) phase, but that the Fe_3Nb_3X phase only contains N. The fraction Nb and N in the Fe_3Nb_3X precipitates is calculated to be 0.51 ± 0.05 wt.% Nb and 197 ± 19 ppm N. The Nb(C,N) precipitates are responsible for 0.016 ± 0.002 wt.% Nb and 15 ± 2 ppm (C,N). For Nb, this would sum up to 0.53 ± 0.5 wt.% precipitated. The amount of N precipitated ends up between 197 ± 19 and 218 ± 21 ppm, depending on how much N the Nb(C,N) phase contains. According to Table 1, only 0.453 wt.%Nb and 170 ppm N is present in the steel. The most logical reason for the calculated excess in Nb and N is again that the fraction precipitates as measured from the BSE images is slightly overestimated. It is therefore better to start from the amount of N in the alloy and to calculate from this the amount of Nb in the Fe_3Nb_3X and Nb(C,N) precipitates, while keeping the ratio of their volume fractions as before. As it is unknown how N is divided between the Fe_3Nb_3X precipitates and the Nb(C,N) precipitates, two limit cases can be defined. If all 170 ppm N has precipitated as Fe_3Nb_3X and the observed Nb(C,N) precipitates would be assumed to contain no N, then 0.44 wt.% Nb would be consumed. If 170 ppm N has precipitated both as Fe_3Nb_3X and as NbN, leaving all C in solid solution in the matrix, 0.43 wt.% Nb would be consumed. As the AES spectrum indicates that the truth lies in between the latter two cases, the precipitated amount of Nb should lie in between these two values, which is a fairly narrow range. Making a mass balance, the (Fe,Cr) matrix contains then 0.01–0.02 wt.% Nb, 0 ppm N and 70–85 ppm C in solid solution.

The precipitation behavior observed in our experiments agree very well with the observations of Fujita who investigated a Fe–19.6Cr–0.78Nb–0.2Si–0.3Mn–0.014C–0.016N steel [9]. The amount of Nb(C,N) precipitates initially present in this steel decrease with heat treatment time at 950 °C and after 100 h the main fraction precipitates is identified as Fe_3Nb_3C. From the chem-

Table 2
Elemental composition of the Fe_3Nb_3X precipitates as measured with WDS.

element	Nb	Fe	Cr	Si	O	N	C
wt.%	60.4 ± 0.2	29.0 ± 0.2	4.8 ± 0.2	3.5 ± 0.1	0.3 ± 0.1	2.3 ± 0.4	0.19 ± 0.02
at.%	41.0 ± 0.6	32.7 ± 0.6	5.9 ± 0.3	7.9 ± 0.2	1.2 ± 0.4	10.4 ± 1.5	1.0 ± 0.1

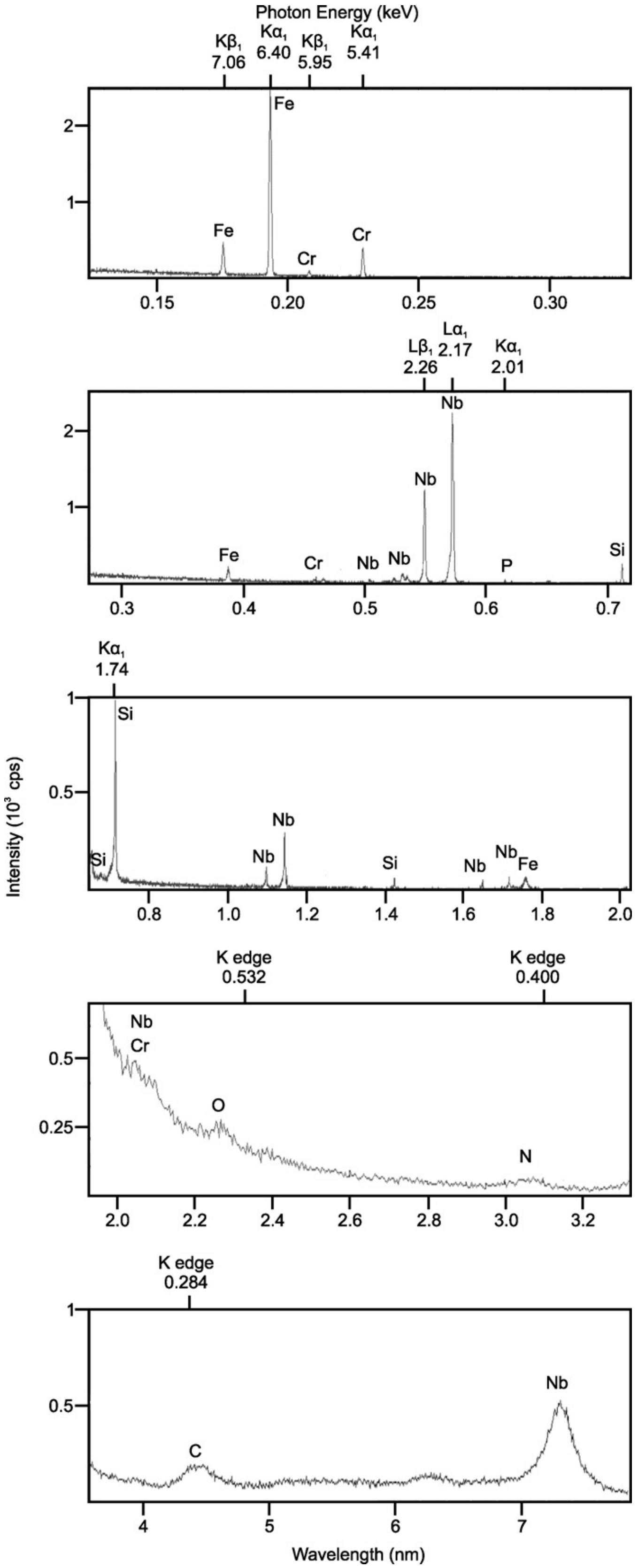

Fig. 4. WDS spectra of the Fe_3Nb_3X precipitates after 500 h at 950 °C in the Nb stabilized ferritic stainless steel.

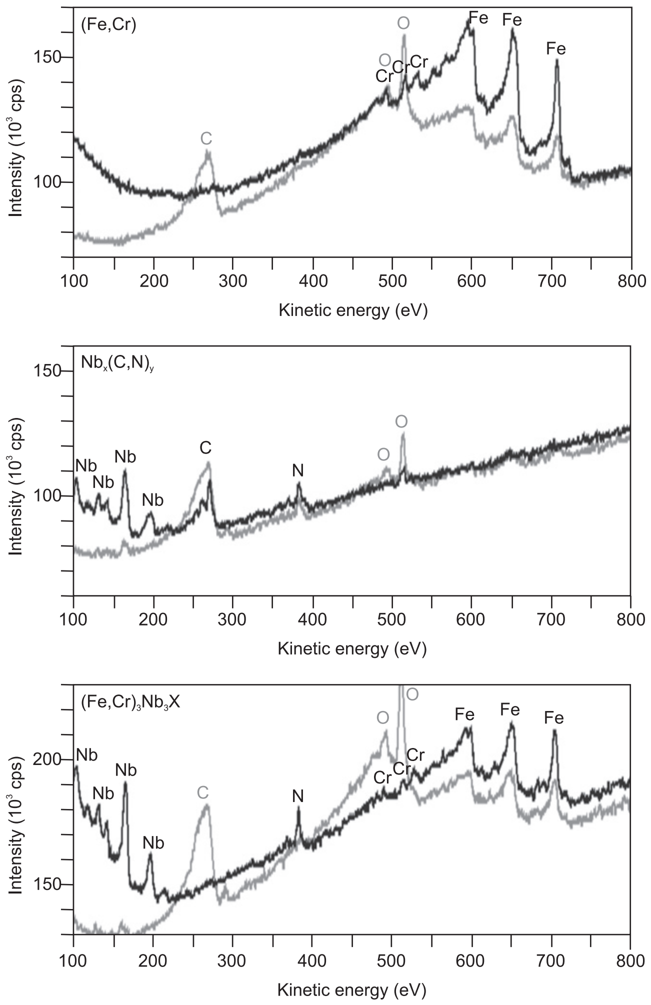

Fig. 5. AES analysis of the (Fe,Cr), $Nb_x(C,N)_y$ and Fe_3Nb_3X phase after 500 h at 950 °C. The spectra in grey are recorded before Ar sputtering, showing clearly C and O peak in all phases. The spectra in black are recorded after 60 s argon sputtering to remove surface contamination. The O peak has clearly decreased in intensity for all three phases, indicating that the presence of this element mainly comes from surface contamination. C is detected in the $Nb_x(C,N)_y$ phase, while N is present in both the $Nb_x(C,N)_y$ and Fe_3Nb_3X phase.

ical analysis of the electrolytically extracted residues, the amount of elements precipitated in the steel is calculated as 0.443 wt.% Nb, 0.234 wt.%Fe, 0.041 wt.% Cr and 0.014 wt.% N. From this it is deduced that most N is precipitated. It is assumed that the N remains present as NbN during the heat treatment, while the NbC precipitates dissolve to form Fe_3Nb_3C. However, based on the amount of elements in the extracted residue, another possibility can be proposed based on the WDS analysis of the Fe_3Nb_3X precipitates from this study. Assuming that the 0.014 wt.% N precipitated as Fe_3Nb_3N and not as NbN, this would lead to 0.36 wt.% Nb precipitated as Fe_3Nb_3N. The remaining 0.42 wt.% Nb will partially react with C to NbC. Assuming that all 140 ppm C will precipitate, about 0.11 wt.% Nb would be precipitated as NbC. This would give a total of 0.47 wt.% Nb precipitated, which is close to the value measured in the extracted residue.

Other works in which precipitation of the Fe_3Nb_3C phase has been described all mention a certain amount of N [4,6]. It is not our purpose to claim what the exact nature of the Fe_3Nb_3X precipitates in these studies is. We want to draw the attention to the possibility that not C, but N is stabilizing these Fe_3Nb_3X precipitates. One might be doubtful about the role of C as an effective stabilizer of the Fe_3Nb_3X precipitates, considering the fact that Lü and Jack were able to compose the oxide Fe_6Nb_6O and nitride Fe_3Nb_3N, but not the equivalent carbide [8]. However, as the investigated steels are all multicomponent systems, it is not impossible that one of these alloying elements has a positive effect on the stabilization of the Fe_3Nb_3X phase through either C or N thereby enabling the precipitation of the nitride or carbide in the steel. It is nonetheless not right to assume that it is a carbide or nitride without analyzing the light elements in these Fe_3Nb_3X precipitates. We have investigated the nature of these Fe_3Nb_3X precipitates by quantitative elemental analysis thereby revealing the presence of the nitride Fe_3Nb_3N in Nb stabilized ferritic stainless steel at high temperature.

This investigation shows that for the design of Nb stabilized ferritic stainless steel grades the N concentration should be taken into account for applications around 950 °C. More precisely, the available N content is important. The concentration of available N can be lower than the nominal content, when other elements that form very stable nitrides, such as Al or Ti, are added to the steel. For the investigated steel, the precipitation of Fe_3Nb_3N decreases the concentration of Nb in solid solution in the (Fe,Cr) matrix to very low values. It can be expected that this will negatively influence solid solution strengthening by Nb at 950 °C. On the other hand, the intergranular precipitation of Fe_3Nb_3N could enhance grain boundary pinning and grain boundary creep resistance. The effect of the Fe_3Nb_3N precipitates on the high temperature mechanical properties of Nb stabilized ferritic stainless steel grades is however not within the scope of this investigation.

5. Conclusions

The formation and nature of the Fe_3Nb_3X precipitates of the Fe_3W_3C type observed after heat treatment at 950 °C of a Nb stabilized ferritic stainless steel containing 82 ppm C and 170 ppm N is investigated with SEM, TEM (SAED+EELS), WDS and AES. The Fe_3Nb_3X phase precipitates mainly at grain boundaries. The volume fraction increases with heat treatment time until the equilibrium fraction is reached. Precipitate coarsening is observed in the sample heat treated for 500 h. AES analysis reveals that these Fe_3Nb_3X precipitates are stabilized by N and not by C and O. The presence of N is confirmed by EELS for small precipitates. The amount of N in large Fe_3Nb_3X precipitates is measured with WDS as 10.4 at.%. For the first time in Nb stabilized ferritic stainless steel, the nature of the element X in the Fe_3Nb_3X precipitates is identified and quantified with multiple techniques. This methodology has revealed that the Fe_3Nb_3X precipitates can be stabilized by N although former studies only take into account the stabilization by C.

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