Electrochemical extraction of microalloy carbides in Nb-steel[(•)]

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Abstract

An electrochemical extraction technique was evaluated as a characterization method of precipitates in microalloyed steels. To carry out this study, a fully precipitated Nb-modified SAE 8620, was used. The investigation was accomplished by varying the electrolyte composition and the filter pore sizes used to collect the particles. The quantification of Nb was performed by inductively coupled plasma-atomic emission spectroscopy (ICP-AES). Microscopy examination was also performed on extraction replicas and on residues collected in the acetate filters. Additionally, X-ray diffraction analysis (XRD) on the collected residues containing the precipitates was accomplished. The results showed that the dissolution rate is faster in the electrolyte consisting of 10 vol% HCl in water. Additionally, transmission electron microscopy (TEM) analysis along with chemical analysis by ICP-AES, showed that (Nb,Ti)C particles were collected in acetate filters with a pore size much larger than the precipitate size.

Keywords

(Nb,Ti)C precipitates; Electrochemical extraction; Steel matrix dissolution; Microalloyed steel; Anodic dissolution; Filtration.

Extracción electroquímica de carburos de microaleación en Nb-acero

Resumen

La técnica de extracción electroquímica fue empleada para evaluar su efectividad en la caracterización de precipitados en aceros microaleados. Para realizar el estudio, se utilizó un acero SAE 8620 modificado con niobio. La investigación se llevó a cabo variando la composición del electrolito utilizado en la disolución electroquímica de la matriz y el tamaño de poro de los filtros usados para recolectar las partículas precipitadas. La cuantificación del contenido de Nb se hizo por espectroscopia de emisión atómica de plasma (ICP-AES). Además, las muestras obtenidas por réplicas de extracción y las provenientes de los residuos recolectados en los filtros fueron analizadas mediante microscopía electrónica de transmisión. Estas últimas muestras, también fueron caracterizadas por difracción de rayos X. Los resultados mostraron que la velocidad de disolución electroquímica de las muestras es mayor en el electrolito acuoso con 10 vol. % HCl. Adicionalmente, el análisis mediante microscopía electrónica de transmisión, en combinación con los análisis químicos mediante ICP-AES, mostró que las partículas de (Nb,Ti)C son recolectadas en filtros de acetato con tamaños de poro mucho mayor que el tamaño de las partículas precipitadas.

Palabras clave

(Nb,Ti)C precipitados; Extracción electroquímica; Disolución electroquímica de la matriz de acero; Aceros microaleados; Disolución anódica de matriz de acero; Filtración.

1. INTRODUCTION

The mechanical properties of microalloyed steels are strongly influenced by the nature of precipitated particles, with the type, volume fraction and particle size distribution being prominent factors determining engineering performance. These particles can affect the recrystallization behavior, subsequent grain growth of the austenite, and induce precipitation hardening in the final matrix. There are numerous studies on the precipitation of carbides in microalloyed steels using both experimental and modeling approaches[1-7]. Most of the experimental investigations have focused on the identification of carbide types, precipitation kinetics, particle coarsening behavior and particle size distributions for different alloy additions and processing conditions,

providing an understanding of the interrelationship of precipitated particles with recrystallization and grain growth phenomena[8-13]. The results have contributed to the development of steels with superior properties by adjusting compositional and thermomechanical variables. Past studies to characterize precipitation in microalloyed steels have been mainly carried out using transmission electron microscopy (TEM) of extraction replicas and thin foils[14-17]. However, quantitative chemical analysis is also helpful to determine the amounts of microalloying elements in solid solution or contained in precipitates. Such investigations can be performed using chemical or electrochemical procedures, selectively dissolving the steel matrix, and separating the undissolved particles from the matrix by filtration. Chemical extraction is a relatively simple method to separate the particles from the matrix, but because

of the strong acid solution concentrations, small particles can also be lost via dissolution[18].

Some important strengthening precipitates in microalloyed steels are typically in the 5 to 20 nm size range[17]. Electrochemical dissolution procedures in this range seems to be the best choice, since more diluted solutions are used that are less likely to dissolve fine particles. This procedure involves anodic dissolution of the steel matrix in an aqueous or non-aqueous electrolyte, followed by the collection of undissolved particles in special filters. Potentiostatic methods have been commonly applied to aid dissolution[19]. The main variables involved in these processes are the composition of the electrolyte used, and subsequent treatment of the residues after particle separation from the steel matrix. Most of the electrolytes used for steels that have been published in the literature are listed in Table I [18 and 20-30], along with information

Table I. Electrolytes used for particle extraction, along with experimental conditions and steels investigated

Tabla I. Electrolitos usados para extracción de partículas, junto con las condiciones experimentales y acero sinvestigados

Electrolytes	Steels (wt% of element)	References
5 % KBr-6 % citric acid-water	Nb-Ti 0.04 % C and 0.07 % C microalloyed steels	[18]
10 % HCl-water 6N HCl-water	1483, 1497 and 1556 Steels:C-Nb steels with different N contents	[18]
10 % Na-citrate-1.2 % KBr-0,5 % KI-water	1483, 1497 and 1556 Steels:C- Nb steels with different N contents	[18]
Methanol-10 % maleic acid, 2 % tetramethyl	0.0068C –0.038Al-0.026Ti and 0.082Ti ammonium chloride	[20]
6N HCl-water	0.003 % C -7 % Cr-0.4 % V-0.09 % N steel Nb-V-Ti HSLA steels	[21] [22]
1 % tetramethyl ammonium 10 % Acetyl	5.Nb-Ti bearing HSLA steel acetone chloride-89 % methanol	[23]
5 % KBr-4 % citric acid	0.041 % C to 0.079 % C Nb-Ti microalloyed steels	[24]
5 % Na citrate 2 % KBr 20% phosphoric acid-water NaCl-KBr-water HCl-citric acid water	1040 and 4340 steels	[25]
HCl—alcohol	18 % Ni maraging steel with and without Ti	[26]
10% HCl- ethyl alcohol –Glycerine	347 austenitic stainless steel	[27]
45 % ferric chloride-water	Tempered low alloy steels (0.35%C-0.77Mn-0.06Ni-0.51%Cr)	[28]
3 % $FeCl_3$-7 % HCl-ethylene glycol	0.05C to 0.059C-0.08Nb, 0.03 Mo-0.29Mo -0.022Al to 0.047Al-0.0059 to 0.0102N	[29]
Neutral Na –citrate solution CdI_2	SAE 1045 tempered for 24 hours at different temperatures	[30]

of the experimental conditions employed and steels investigated. Residues collected in the filters can be directly analyzed, depending on the analytical technique selected. Some researchers use X-ray fluorescence analysis (XRF) of the residues, but recently, inductively coupled plasma atomic emission spectroscopy (ICP-AES) and inductively couple plasma optical emission spectroscopy (ICP-OES) have been more commonly used. In order to perform the chemical analysis by ICP-AES or ICP-OES, the particles collected in the filters must be converted into a soluble form. Three methods have been used for that purpose, including digestion, fusion and acid dissolution. For the digestion process, the filters with their residues are sealed in a pressure vessel, mixed with selected acids and heated. Different combination of acids can be used. In one report, $HCl/HNO_3/HF$ in a volumetric ratio of 3:1:0.5 was employed[31]; in another work, HF/HNO_3 in a ratio of 1:5 [32] was used. Fusion is accomplished by mixing the residues with a salt. The mixture is then heated above the salt's melting point where the particles are dissolved. Various salt combinations for fusion have been utilized. H. Jo Jun *et al.*[23], used a mixture of three parts of Na_2CO_3 and one part of H_3BO_3, and M. Saeki *et al.*[19] suggest the use of sodium pyrosulfate. In the acid dissolution procedure, appropriate combinations of concentrated acids are utilized.

Even though several investigations have used the electrochemical method to extract particles in steels, detailed procedures and difficulties involved in the process are not well documented. The aim of this work was to perform a systematic evaluation of a Nb-microalloyed carburized steel, using electrochemical extraction of carbides. Polarization curves, rates of steel matrix dissolution, type and size distribution of the carbides collected in the filters, and filter pore sizes used, are reported.

2. EXPERIMENTAL PROCEDURE

2.1. Material and heat treatments

The experimental steel used in this study was a Nb-modified SAE 8620 grade, with a nominal chemical composition given in Table II. The laboratory-melted steel was cast as an ingot, reheated to 1,230 °C, forged, and air-cooled to room temperature. The billets where then rolled after reheating at 1,260 °C for approximately one hour. The thermomechanical procedure has been described in detail elsewhere[33]. Specimens 70 mm x 18 mm x 1.5 mm of this material were sealed in evacuated and argon back-filled quartz tubes, solution treated at 1,300 °C for 30 min, followed by iced-water quenching. Precipitation heat treatments were then carried out at 700 °C for 2 h and 72 h. Additionally, some of the specimens subjected to precipitation heat treatments at 700 °C for two hours, were heated to 1,050°C, held for one hour at this temperature and quenched in water to dissolve the cementite particles. Rectangular samples 15 mm x 10 mm x 1.5 mm were then cut from the heat treated specimens, to perform the electrochemical dissolution tests to isolate the particles from the steel matrix, and quantify the amount of Nb that was in solid solution and or in the form of a precipitate. Preliminary electrochemical dissolution trials were conducted to establish the parameters for particle extraction of carbides. Thus, potentiodynamic and potentiostatic tests using various electrolytes were carried out to guide in the selection of the test conditions. The methodology used for these experiments is described in detail below.

2.2. Electrochemical tests

2.2.1. *Potentiodynamic tests: Polarization curves*

In order to define the appropriate conditions for electrochemical dissolution of the steel matrix, potentiodynamic tests were performed in three of the most commonly used electrolytes[1, 4 and 6-9]: a) 10 vol. % HCl + deionized water, b) 5 % KBr + 6 % citric acid + deionized water; and c) 10 % acetyl acetone + 1% tetramethyl ammonium chloride + methanol. These solutions will be referred to in the text as "A", "B" and "C", respectively. To carry out these experiments, a copper wire was soldered to the back

Table II. Nominal chemical composition of the experimental steel used in this study, in wt. %

Tabla II. Composición química nominal del acero utilizado en este estudio, en porcentaje en peso

C	Nb	Ti	Mn	P	S	Si	Cr	Ni	Mo	Al	N, ppm
0.220	0.054	0.034	0.800	0.017	0.003	0.240	0.600	0.430	0.210	0.022	92

of 15x10x1.5 mm³ specimens. The specimens were then mounted in bakelite, ground through 600 grit, ultrasonically cleaned in methanol and dried. Polarization curves were obtained for each electrolyte, using a standard corrosion cell, two graphite counter electrodes and a standard calomel electrode (SCE).

2.2.2. Potentiostatic tests

To select the preferred electrolytic solution for electrochemical dissolution of the steel matrix, potentiostatic tests were carried out using the three experimental solutions employed in the potentiodynamic tests. These experiments were run for different times to determine the matrix dissolution rates, using specimens treated at 700 °C for 2 h. To assess the rate of specimen dissolution for each electrolyte, 15 mm x 10 mm x 1.5 mm specimens were accurately weighed to ± 0.0001 g, then electrochemically dissolved at constant potential, and the weight lost monitored with time.

2.3. Transmission electron microscopy (TEM) and X-ray diffraction (XRD) analyses

Samples heat-treated at 700 °C for 2 h and 72 h, were examined by TEM using a Philips CM 200 microscope equipped with energy dispersive X-ray spectroscopy (EDS). Characterization of the bulk samples was carried out on carbon extraction replicas prepared after polishing and etching in 2 % nital for 2 s. After carbon film deposition, the samples were submerged in 10 % nital for a maximum of five minutes to strip the carbon film. The films were then rinsed five times in a solution of 50 vol. % methanol in water. Fresh solutions were used in each step for each sample. From this analysis, the niobium carbide particle size distributions were obtained by measurements of about 200 particles.

Residues collected in filters, following electrochemical particle extraction procedures, were also examined by TEM. This analysis was conducted to evaluate the size of Nb-precipitates collected during filtration upon a sample first treated at 700 °C and then heated to 1,050 °C and held for one hour, to dissolve any iron carbides formed at 700 °C. The residues were directly collected in a 0.22 mm cellulose acetate filter, without any previous filtration step. After filtering, the residues were rinsed thoroughly with deionized water, and the cellulose acetate filter was dissolved with ethanol. Two drops of the solution were

deposited on a carbon coated copper TEM grid. Once the specimens were completely dried, the grid was coated again with a carbon film, using an evaporation procedure.

X-ray diffraction (XRD) analysis was conducted on two specimens. The first one heat treated at 700 °C for two hours and the second, treated with the same conditions and then heated at 1,050 °C and held for one hour followed by quenching, to avoid the formation of cementite. In this way, the peaks of cementite were not present in the diffraction pattern, which facilitated the evaluation of other carbides and nitrides. An Anodisc™ filter (0.02 μm pore size) was used to collect the particles of the first specimen, but for the specimen heat-treated at 1,050 °C for one hour, a cellulose acetate filter (0.22 μm pore size) was used because the collected residues for this specimen, once dried, did not adhere to the Anodisc™ filters. The amount of sample electrochemically dissolved to perform the XRD was about two grams. The XRD data were obtained using copper Kα radiation, a scan step of 0.05°, a step time of five seconds and 2q range from 30-80° in a Siemens Kristalloflex 810 diffactometer.

2.4. Chemical analysis

Chemical analyses were performed on heat-treated samples electrochemically dissolved using electrolyte "A" (10 vol. % HCl in deionized water). In order to do it, the samples were first electrochemical dissolved and then filtered to separate the precipitates from the solution. For the filtering process, a series of cellulose acetate filters were utilized, with pores sizes of: 8 μm, 0.45 μm, 0.22 μm, 0.025 μm and 0.010 μm. After each filtration step, filters were rinsed with deionized water, and the filtrate solution was transferred to a 500 ml calibrated flask and diluted with deionized water to volume. Residues collected in each filter were placed in separated beakers and dissolved in concentrated acids. The solution was then transferred to a 100 ml calibrated flask and the volume completed with deionized water. Each solution was analysis by ICP-IES to determine the amount of Nb. High-purity ICP standard solution (1000 ppm) of niobium was utilized. Five different Nb concentrations (0.25 ppm, 2.5 ppm, 5 ppm, 25 ppm and 50 ppm), were selected to obtain the calibration curve shown in figure 1. The standards were prepared using an iron-matrix solution as solvent, obtained by chemical dissolution of a low-carbon Nb-free steel. Specimens in the as-received condition were also electrochemically and chemically dissolved, to determine the total amount of Nb in the steel.
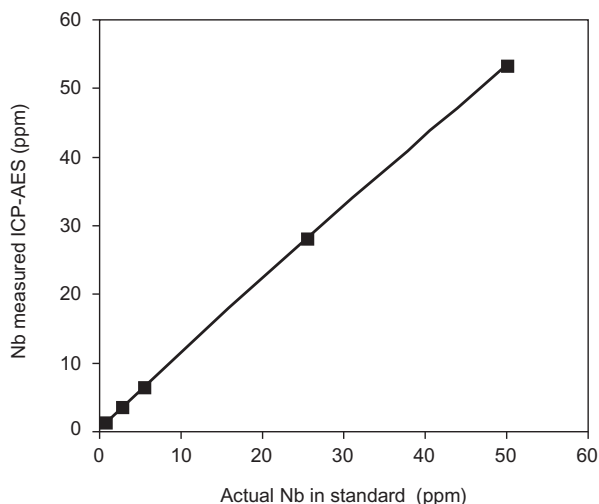
Figure 1. Calibration curve for analysis of Nb by ICP-AES.

Figura 1. Curva de calibración para el análisis de Nb por ICP-AES.

3. RESULTS AND DISCUSSION

3.1. Potentiodynamic and potentiostatic tests

Figure 2 shows current density (I) as a function of applied potential (E), obtained from the potentiodynamic test for the three electrolytes
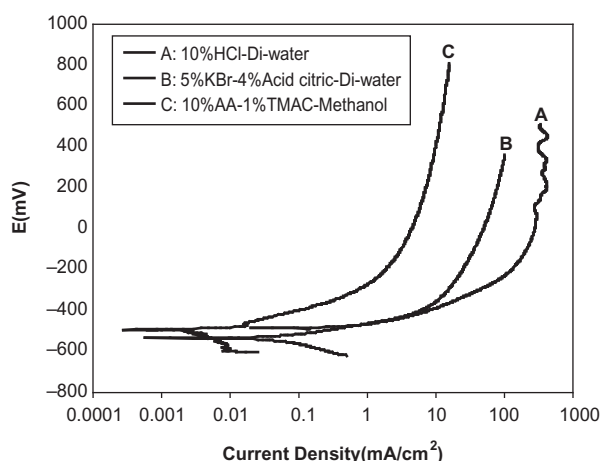

Figure 2. Potentiodynamic curves for the Nb/Ti-modified SAE 8620 steel in three different electrolytes.

Figura 2. Curvas potenciodinámicas para el acero SAE 8620 modificado con Nb utilizando tres electrolitos diferentes.

used in the preliminary experiments. The current density levels for a specific potential are much lower in electrolyte "C", indicating that the dissolution rate is slower in this electrolyte. As a consequence, the amount of material dissolved with time at a specific potential, will be lower for this electrolyte. This result is in agreement with the data obtained from the potentiostatic tests, illustrated in figure 3. This figure shows the amount of steel dissolved with time, at a constant potential. Electrolyte "A" causes a greater dissolution of material for a given time, compared with electrolytes "B" and "C". Taking into account this result, electrolyte "A" was selected for the electrochemical dissolution of the steel specimens. Based on the polarization curves in figure 3 and reports of other researchers [18, 24], the potential selected for anodic dissolution of the steel matrix in solution "A", was −150 mV (…0.15V). At this potential, the current density and dissolution rate are optimized. On the other hand, the dissolution time was taken as eight hours to obtain an amount of sample dissolved of around one gram, which is enough to perform the chemical analysis of Nb by ICP-AES. It is important to point out that to be able to determine effectively the Nb in solution, tartaric acid is added to the HCl aqueous electrolyte to suppress hydrolysis of this element[34].
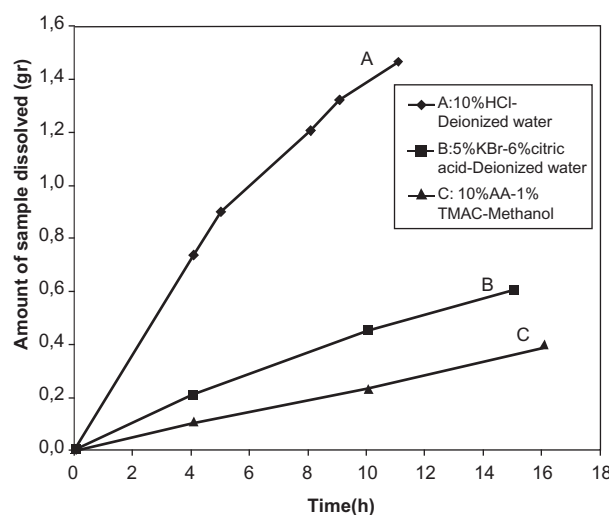

Figure 3. Amount of specimen electrochemically dissolved with time, in three electrolytes, using a potential of -0.15V for electrolytes A and B, and 0.30V for electrolyte C.

Figura 3. Cantidad de muestra disuelta por métodos electroquímicos en función del tiempo en tres electrolitos, utilizando un potencial de -0,15V para los electrolitos A y B y 0,3V para el electrolito C.

3.2. TEM and XRD analyses of heat treated samples

Figure 4 shows TEM micrographs and EDS spectra of precipitates from carbon extraction replicas of samples heat treated at 700°C for 72 h. In Figure 4A, fine precipitates of rounded morphology are observed. These particles are rich in Nb and also contain some amount of Ti and Mo, as revealed by the EDS spectrum of figure 4B. The presence of Mo in the Nb precipitates has been reported for other researchers[35]. Figure 4C shows larger particles with a square shape. These particles are rich in Ti, as indicated by the EDS spectrum of Figure 4D and may correspond to a titanium nitride. Particle size distributions of the Nb rich precipitates from TEM analysis are displayed in figure 5. The particles have a diameter in the 3 to 9 nm range, and 5 to 13 nm for the samples treated at 700 °C for 2 h and 72 h, respectively. The average size of Nb rich particles increases from 7 ± 3 nm to 9 ± 4 nm when the time at 700 °C was increased from

2 h to 72 h. So the extent of coarsening with time of Nb rich particles was relatively small at this temperature. Low coarsening rates of these particles may be attributable to the Mo segregation to the precipitate/ferrite interface. Molybdenum modifies the interfacial energy and possibly restricts the diffusion of Nb atoms from ferrite to the Nb rich particles, as pointed out by Uemori et al.[35].

Figure 6 shows a diffraction pattern of the residue collected in the Anodisc™ filter for a sample treated at 700 °C for 2 h. Most of the peaks correspond to cementite, while there are other carbide and nitride peaks also present in this specimen. These peaks generally overlap the ones of cementite. The microalloyed precipitate peaks are better revealed on the residues of sample heated at 1,050 °C for one hour, where cementite is not present, as illustrated in figure 7. For this case, cellulose acetate filters were used. The diffraction peaks in this figure match well with (Nb,Ti)C and TiN, with lattice parameters of 0.436 nm and
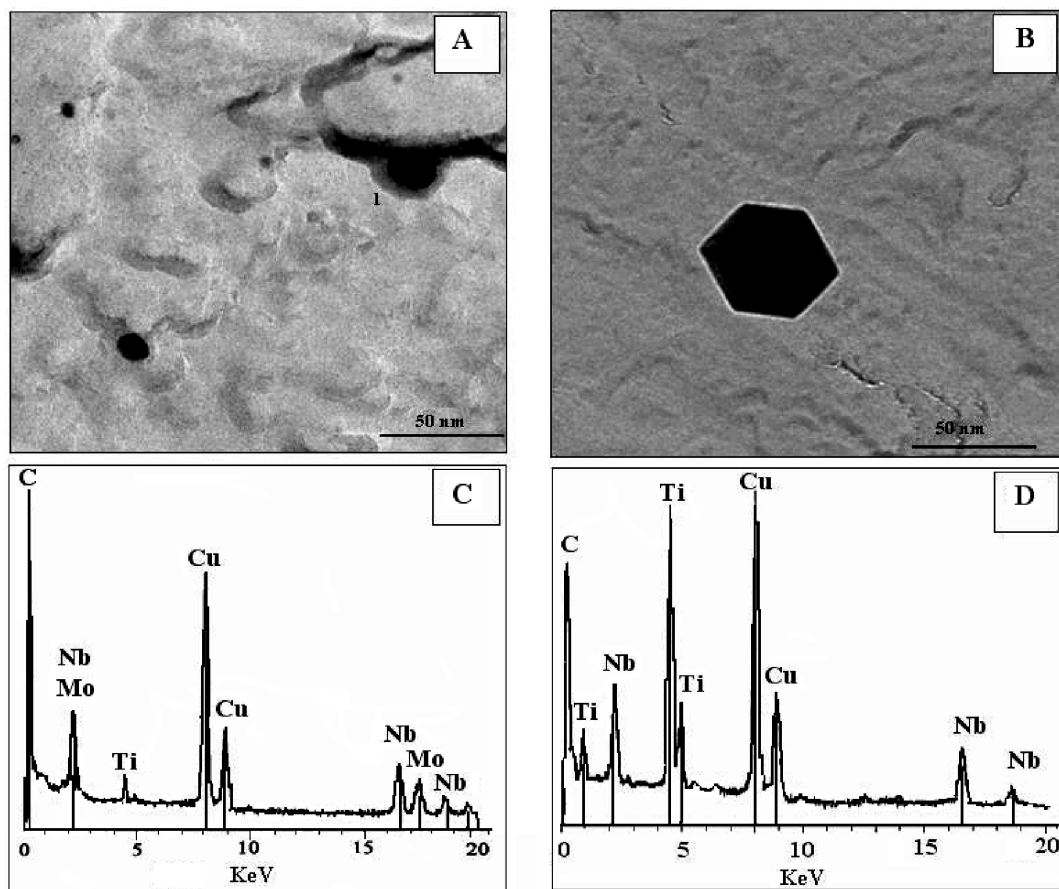

Figure 4. TEM micrographs of the specimen held at 700°C for 72h, with their respective EDS spectrums.

Figura 4. Fotomicrografías por TEM de muestras mantenidas a 700°C por 72 h con sus respectivos espectros de EDS.
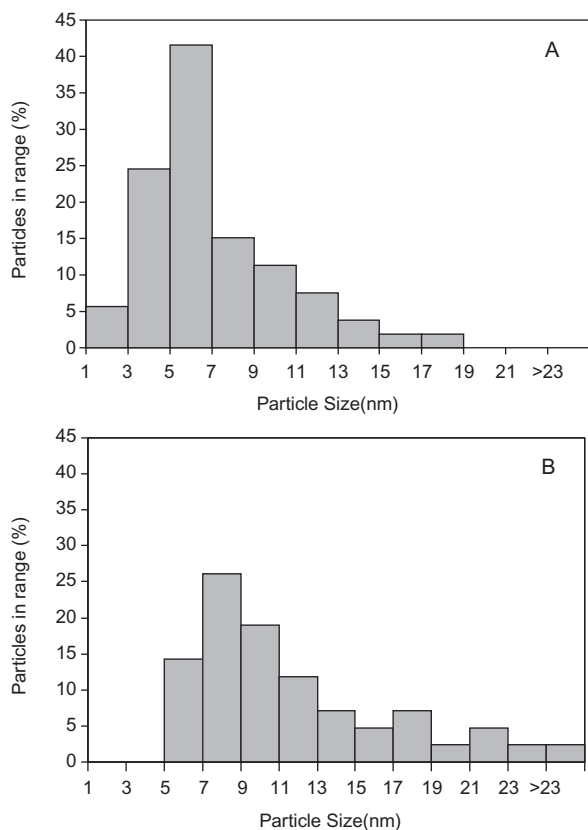
Figure 5. Particle size distributions in specimens heat treated at 700°C for A) 2 h and B) 72 h.

Figura 5. Distribución de tamaño de partícula para las muestras tratadas a 700°C A) 2h y B) 72h.
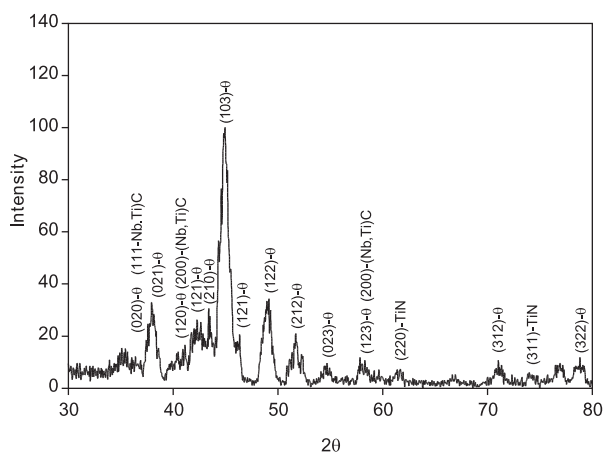

Figure 6. X-ray diffraction pattern of residues collected in "Anodisc^{TM}" filter (20 nm pore size) for a specimen held at 700°C for 2 h.

Figura 6. Patrón de difracción de rayos X de los residuos recolectados en un filtro "Anodisc^{TM}" (tamaño de poro de 20 nm) para muestras tratadas a 700°C durante 2h.
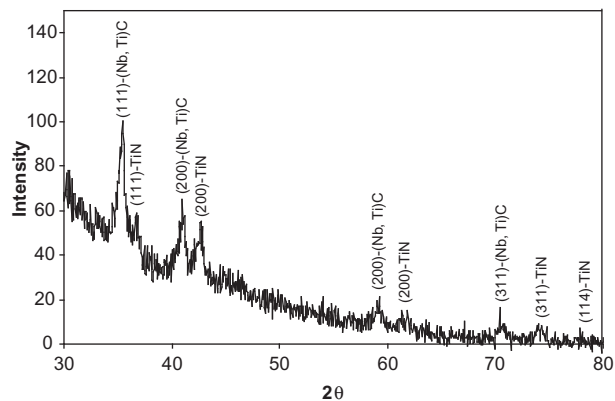

Figure 7. X-ray diffraction pattern of residues collected on a cellulose filter (200 nm pore size) for a specimen held at 700°C for 2h, then heated at 1050°C, held for 1h and water quenched.

Figura 7. Patrón de difracción de rayos X de los residuos capturados en un filtro de celulosa (tamaño de poro 200 nm) para una muestra mantenida a 700 °C por 2h, luego calentada a 1050 °C durante 1 h y templada en agua.

0.426 nm, respectively. Therefore, the fine and coarse precipitates rich in Nb and Ti, respectively, observed in figure 4, correspond to these two types of precipitates. The results can be explained considering that titanium nitride precipitates are extremely stable. From solubility product [37], the solubility temperature of TiN for the steel under investigation, is estimated to be about 1800°C. In consequence most of the Ti ($\cong 0,0315$ wt %) may be combined with N as TiN, while the excess (0,0025 wt %), formed (Nb,Ti)C, since the amounts of Ti and N in the steel under study, are almost in a stoichiometric ratio.

3.3. Chemical analysis and characterization of residues by TEM

Quantitative chemical analysis of Nb was performed by ICP-AES on samples in the as-received condition and on specimens held at 700 °C for different times electrochemically dissolved. The results are displayed in figure 8. The total amount of Nb in the steel is 623 ppm, from which 483 ppm of it was in a precipitate form and an apparently 140 ppm was in solid solution. As a reference, the total amount of Nb was also determined in samples chemically dissolved, obtaining a similar result (645 ppm). The ICP analysis for samples held at 700 °C for two hours showed that Nb precipitated in the amount of 471 ppm, while it was
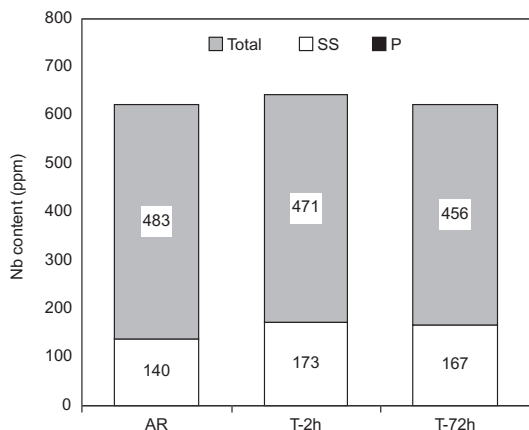
Figure 8. Nb in solid solution (SS) and precipitated (P) in samples in the as-received condition (AR), and samples treated at 700 °C for 2h (T-2h) and 72h (T-72h).

Figura 8. Contenido total de Nb en solución sólida (SS) y precipitado (P) en muestras en condición de recibido (AR) y en muestras tratadas a 700 °C por 2h (T-2h) y por 72h (T-72h).

456 ppm in the one held for 72 h. The apparent Nb levels in solution for both samples were 173 ppm and 167 ppm, respectively. It is important to point out that the Nb mass balance, considering the sum of the

Table III. Apparent Nb in solid solution (Nb SS), Nb precipitated (Nb P) and total Nb (Total Nb= Nb SS+ Nb P) measured by ICP-AES in six samples treated at 700 °C for 2 h and the average and standard deviation values.

Tabla III. Cantidad de Nb en solución (Nb SS), Nb precipitado (Nb P) y Nb total (Nbtotal= Nb SS + Nb P), con sus desviaciones standars (STD), determinadas mediante ICP-AES en 6 muestras tratadas a 700°C por 2h.

	Nb SS (ppm)	Nb P (ppm)	Total Nb (ppm)
	182	480	662
	144	483	627
	167	481	648
	136	464	600
	155	513	668
	160	455	615
Average	157	479	637
Standard deviation	16	20	27

Nb precipitated and Nb in solid solution, matches well with the total Nb content in the steel. To assess the reproducibility of the chemical analysis data, the Nb levels in solution and precipitates were measured in six additional samples held at 700 °C for two hours. The results are reported in table III, with the average levels of apparent Nb in solution at 157 ±16 ppm, and Nb precipitated at 637±27 ppm. Considering the typical standard deviation of ICP-AES data, the difference in the amount of Nb precipitated between the samples held at 700 °C for 2h and 72 h (Fig. 8) is not significant. This observation is in accordance with data reported by other researchers[18].

A thermodynamic calculation using the solubility product of NbC in ferrite, given by equation 1 [4], indicates that at 700 °C, the equilibrium amount of Nb in solid solution in the steel under study (0.06 wt.% Nb and 0.2 wt.% C), should be about 74 ppm. The Nb in solution obtained by ICP-AES is 159 ±16 ppm. The excess Nb in solution measured in these samples may be due to dissolution of small particles during anodic dissolution and/or by the loss of some small (Nb, Ti)C particles during filtration. Taking into account the limited amount of Nb that was retained in filters of small pore sizes (Fig. 9), it seems more likely that some small particles may have dissolved during electrochemical dissolution of the steel matrix. Future work is needed to examine these effects, but the method described here should be especially useful in studies when precipitates form at higher temperatures and are large enough to resist electrochemical dissolution.
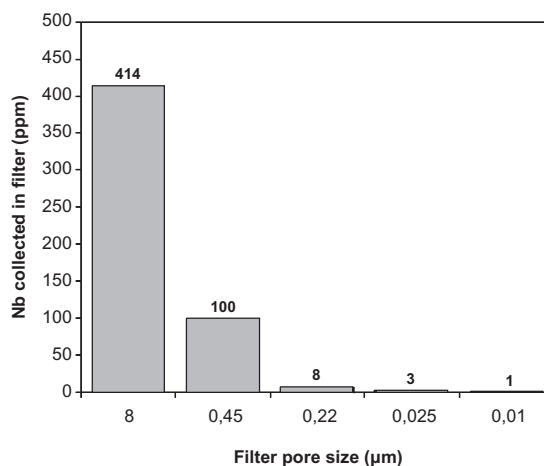

Figure 9. Content of Nb (ppm) in the residues collected in cellulose acetate filters with different pore sizes.

Figura 9. Cantidad de Nb en los residuos recolectados en los filtros de acetato con diferentes tamaños de poros.

$$\log_{10}[\text{Nb}][\text{C}] = 5.43 - \frac{10960}{T} \quad (1)$$

The amount of Nb in the residues collected in each of the filter with different pore sizes was also determined, and the results are presented in figure 9. Most of the Nb particles were retained in the 8 mm filter and practically no Nb particles were left for collection in the filters with the smallest pore sizes (0.025 μm and 0.010 μm). Similar results were obtained in other studies[18], and are in agreement with the TEM results displayed in figure 10. This figure shows (Nb,Ti)C particles collected in a 0.22 mm cellulose acetate filter for the sample tempered at 700 °C for two hours and then heated and held at 1,050 °C for 1 h. The particle sizes in figure 10 are in a range of 5 to 55 nm (0.005 μm-0.055 μm), which is smaller than the filter pore size. This result can be explained by electrostatic attraction effects; according to Kroger et al.[36] dielectric filters accumulate a significant amount of charge, which is mainly concentrated at the vicinity of the pore rims. Another possible explanation, is the agglomeration of particles that may occur because of the small size of precipitates[18].
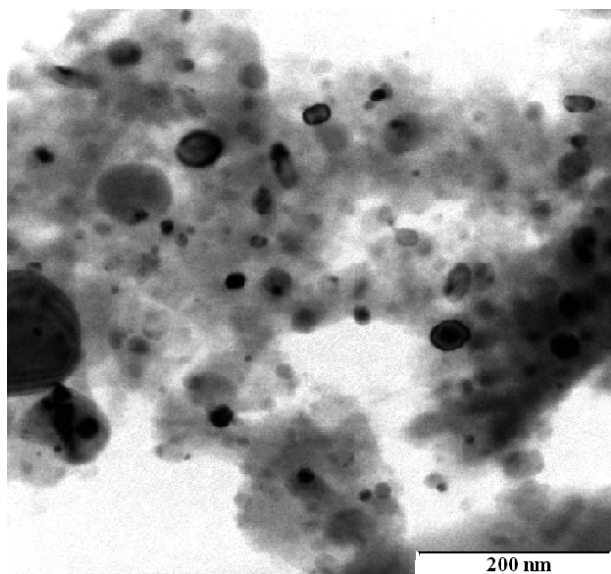

200 nm

Figure 10. TEM micrograph of residues collected in the 0.22 μm cellulose acetate filter pore size, of sample heated and hold at 1050 °C for 1h.

Figura 10. Fotomicrografía por TEM de los residuos recolectados en un filtro de acetato con tamaño de poro de 0,22 μm, correspondiente a una muestra calentada y mantenida a 1.050 °C por 1h.

CONCLUSIONS

The precipitation in a Nb-modified carburizing steel was determined by applying an electrochemical matrix dissolution method to samples selectively heat treated to obtain the precipitation. An electrolyte containing HCl is effective to dissolve the steel matrix. Consistent results have been obtained with this procedure, using 8 h of electrochemical dissolution and –0.15 V, although some fine particles might be dissolved during this process. The technique, which involves filtering solutions through filters of know pores sizes, showed that particles were captured in filter with greater pore size than the average particles size, which has been associated to the effects of electrostatic attraction and agglomeration of small particles. Two types of phases containing Nb were identified in the samples: fine (Nb,Ti)C with some Mo segregation; and, coarse (Ti,Nb)N precipitates which did not dissolve during full dissolution heat treatment.

Acknowledges

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