

Determination of niobium in Nb-stabilized stainless steel by ICP-AES technique

Abstract

Niobium is a very useful alloying element in the steel industry owing to its different applications. One of the important applications is the use of niobium in the stabilization of austenitic and ferritic stainless steel. Owing to excessive carbon content in these steels, formation of chromium carbide takes place along the grain boundaries leading to the formation of a chromium depleted region. Formation of chromium carbide is detrimental and leads to corrosion of the chromium depleted region, which is normally termed as "inter-granular corrosion". Stabilization is a process in which the formation of chromium carbide is avoided by addition of niobium and titanium. Niobium is generally preferred to titanium due to the detrimental effects of titanium such as surface and finishing of the material.

In the present study, analysis of niobium in an Nb-stabilized stainless steel sample was carried out as part of the certification activity organized by the Bureau of Analyzed Samples, (BAS) Ltd, UK. The analysis was carried out by ICP-AES technique and the concentration of Nb was determined to be 0.6006%. The determination of niobium in our laboratory has been discussed in the present study.

Keywords: niobium, ICP-AES, stainless steel, BAS, certification

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Introduction

Austenitic stainless steels (ASSs) are well known for their excellent mechanical properties and are the most corrosion resistant among the stainless steel group.¹⁻³ However, they are susceptible to pitting corrosion in presence of chloride ions. Pitting corrosion or pitting is a localized type of corrosion which leads to the development of small holes in the metal. This form of corrosion is dependent upon the various alloying elements in the stainless steel, microstructure, heat treatment and overall environmental conditions where the material is being used. The corrosion can be prevented by using niobium and titanium as alloying elements in the stainless steel, by a process known as 'stabilization' and the material is described as stabilized stainless steel. In this process, Nb plays the role of an inhibitor and prevents the formation of chromium enriched $M_{23}C_6$ carbides at the grain boundaries, thus increasing the intergranular corrosion resistance and pitting resistance of the ASSs during the process of ageing.⁴ Different studies have also revealed that as an alloying element, niobium can promote passivation by virtue of incorporation of Nb^{5+} in the passive film, thus retarding global or localized corrosion in ferritic stainless steel,⁵ low alloy steel,⁶ and Ti-based alloy.⁷ Therefore, it can be well understood that Nb is a very important alloying element in stainless steel.

These different studies indicate the usefulness of Nb as an important alloying element and therefore it is important to monitor the percentage of Nb in the stainless steel. The present paper is related to this aspect and describes the determination of Nb in a stainless steel sample BCS/SS-CRM 479 provided by Bureau of Analysed Standards (BAS, UK) for certification purpose. A total of 29 laboratories have participated in the certification program of this sample. The present paper discusses the analysis of Nb in this sample in the subsequent sections.

Experimental

Reagents required

Concentrated hydrochloric acid (HCl), concentrated nitric acid (HNO_3), 40% hydrofluoric acid (HF) were procured from M/s. Rankem, India. Niobium (1000ppm) elemental solution was procured from M/s. Accustandard. Double distilled water (DD) confirming to Type II of ASTM D1193-06.⁸ was used in the preparation of the reagent solutions.

Apparatus required

Polytetrafluoroethylene (PTFE) beaker (250ml) with a PTFE cover was used for sample preparation. PTFE volumetric flask (100ml) was used for making up the sample solutions. All PTFE wares used were of Tarsons make. Spectro make CIROS ICP-AES instrument was used for the analysis of the samples.

Preparation of calibration solutions

A 100ppm working solution was prepared from a 1000ppm niobium elemental solution and was suitably diluted in 100ml volumetric flasks to obtain the following calibration solutions.

To maintain the matrix of these calibration solutions with that of the test sample solutions, 5ml of concentrated nitric acid and 15ml of concentrated hydrochloric acid was added to each of these calibration solutions respectively.

Preparation and analysis of sample solution

The sample analysis was done in quadruplicate. 0.2g of sample was accurately weighed in a precision balance and carefully transferred to a PTFE beaker. 5ml of DD water was added which was followed by addition of 5ml of HF acid. This solution was then subjected to gently

hand stirring for 5 minutes for homogenization of the solution. Then, 5ml of HNO_3 was added followed by addition of 15ml of HCl and again the beaker was gently stirred for homogenization. The beaker was then put on a hot plate on low flame for effective dissolution of the sample. It was observed that in about 10-15 minutes the entire sample was completely dissolved. The resultant solution was then transferred carefully to a 100ml PTFE volumetric flask and the solution was made up to mark after the solution attained room temperature. This solution was then analyzed in the ICP-AES. A CRM sample 237/1 was also prepared in a similar manner as described above to check the efficiency of the method.

The prepared sample solutions were then run in the method made in the ICP-AES by choosing wavelength 309.418nm as the analytical line and taking monitor line as argon (430.10nm). The analytical method was calibrated by using the calibration solutions described in Table 1 above.

Table 1 Details of calibration solutions used for analysis of Nb

Concentration in ppm					
Element	Standard	Standard	Standard	Standard	Standard
	0	1	2	3	4
Niobium	0	2	5	10	50

Results & discussion

The analysis of the sample solutions in quadruplicate was as follows. The result was validated by analyzing a certified reference material (CR) CRM 237/1 and the results are as under (Table 2).

Table 2 Analysis results for Nb in BCS/SS-CRM 479

Result id	Nb (%)
1	0.6001
2	0.6007
3	0.6009
4	0.6006
Average	0.6006
Standard Deviation	0.0003

It was observed that the approximate composition of niobium provided by BAS, UK was 0.6% which was in excellent agreement with the obtained average value. Also, the obtained value and certified value for ECRM 237/1 was in close agreement thus, validating the analysis result for Nb in BCS/SS-CRM 479. The quadruplicate analysis and the average value for Nb was sent to BAS, UK for completing the certification activity (Table 3).

Table 3 Analysis result for Nb in ECRM 237/1

Result id	Nb (%)
Obtained value	0.652
Certified value	0.66

Conclusion

Niobium in stainless steel was analyzed by using ICP-AES for the purpose of the certification of proposed CRM BCS/SS-CRM 479 sent by BAS, UK. The obtained value was in excellent agreement with the approximate composition provided by BAS, UK and the result was further validated with the analysis of ECRM 237/1. BAS UK has included the average analysis result in the certificate issued for this new reference material.

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Conflict of interest

Author declares that there is no conflict of interest.

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