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Original Article

Analysis of Zirconium and Nickel Based Alloys and Zirconium Oxides by Relative and Internal Monostandard Neutron Activation Analysis Methods

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Background: The chemical characterization of metallic alloys and oxides is conventionally carried out by wet chemical analytical methods and/or instrumental methods. Instrumental neutron activation analysis (INAA) is capable of analyzing samples nondestructively. As a part of a chemical quality control exercise, Zircaloys 2 and 4, nimonic alloy, and zirconium oxide samples were analyzed by two INAA methods. The samples of alloys and oxides were also analyzed by inductively coupled plasma optical emission spectroscopy (ICP-OES) and direct current Arc OES methods, respectively, for quality assurance purposes. The samples are important in various fields including nuclear technology.

Methods: Samples were neutron irradiated using nuclear reactors, and the radioactive assay was carried out using high-resolution gamma-ray spectrometry. Major to trace mass fractions were determined using both relative and internal monostandard (IM) NAA methods as well as OES methods.

Results: In the case of alloys, compositional analyses as well as concentrations of some trace elements were determined, whereas in the case of zirconium oxides, six trace elements were determined. For method validation, BCS-certified reference material 310/1 (a nimonic alloy) was analyzed using both relative INAA and IM-NAA methods.

Conclusion: The results showed that IM-NAA and relative INAA methods can be used for nondestructive chemical quality control of alloys and oxide samples.

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1. Introduction

The chemical characterization of materials is an important step in chemical quality control (CQC) exercises. It involves the determination of major, minor, and trace concentrations of elements with good accuracy and precision. CQC helps in ensuring the quality of the material as per the required specifications. In the case of nuclear reactor materials, the finished products should meet the stringent chemical specifications for major to trace constituents for better performance of a nuclear reactor during its operation. The materials of interest are mainly nuclear reactor structural materials of alloy and oxide matrices. It is important to use suitable analytical techniques to analyze these complex matrix samples to obtain good quality results. The routinely used analytical methods are mostly wet chemical and spectroscopic techniques, such as atomic absorption spectroscopy, inductively coupled plasma optical emission spectroscopy (ICP-OES), and ICP-mass spectrometry (ICP-MS), as well as chromatographic techniques. Although these techniques give high quality results, they are destructive methods and are associated with a cumbersome dissolution process that uses various acids. In some cases, radioanalytical techniques such as X-ray fluorescence (XRF), instrumental neutron activation analysis (INAA), prompt gamma-ray NAA (PGNAA), and ion beam analysis are used. Nuclear analytical techniques, such as INAA and PGNAA, have several advantages. They are able to perform simultaneous multielement determination, are nondestructive in nature, have a negligible matrix effect, are highly sensitive and selective, and have achievable high precision and accuracy.

Routine work using conventional INAA is mainly carried out using the relative method, in which *a priori* knowledge of the constituents in a sample needs to be known. By contrast, k_0-based NAA uses a single comparator such as gold (^{197}Au) or any other suitable element that is coirradiated with the same neutron flux along with the sample [1–3]. As the single comparator is different to the element of interest, this method requires neutron spectrum parameters, namely, the epithermal neutron flux shape factor (α) and the subcadmium to epithermal neutron flux ratio (f), absolute detection efficiency, and the k_0 factor [4, 5] for concentration calculations. In the case of k_0-based internal monostandard NAA (IM-NAA), an element present in the sample, which is expected to be reasonably homogeneous, is used as the monostandard [6–8]. IM-NAA using *in situ* relative detection efficiency [7, 8] is useful for the analysis of small- as well as large-size (g–kg scale) and nonstandard geometry samples. The k_0-NAA method was adapted in 1995 in our laboratory. Later, in 2003, the IM-NAA in conjunction with *in situ* relative efficiency method was developed. The irradiation positions of APSARA [9] and KAMINI [10] reactors were characterized for f and α values by the cadmium ratio method using multiflux monitors. As IM-NAA gives mass fraction ratios with respect to the internal monostandard, knowledge of the concentration of internal monostandard is required to obtain absolute concentrations of the elements in the sample. When all the major and/or minor elements present in a sample are amenable to NAA, e.g., in zircaloys and stainless steels, IM-NAA is useful for calculating the absolute concentrations by a standard-less approach using the mass balance procedure.

The IM-NAA method was successfully applied for the standard-less analysis of nuclear reactor structural materials such as zircaloys [7, 11] and stainless steels [7, 8, 12]. For validation of the IM-NAA method, BCS certified reference materials (CRMs) 225/1 (low alloy steel) and 466 (austenitic stainless steel) [8, 12, 13] were analyzed. Various analytical techniques have been used for the analysis of alloys and oxides including NAA [14], prompt gamma-ray NAA [15, 16], XRF [17], spectroscopic methods such as ICP-OES [18–21], atomic absorption spectroscopy [22–26], and ICP-MS [27–29].

This research paper deals with the chemical characterization of zirconium and nickel based alloys by IM-NAA and compares results with conventional INAA and ICP-OES. Samples in triplicate analyzed by INAA, IM-NAA, and ICP-OES are nimonic alloys, Zircaloy 2, and Zircaloy 4. Zirconium oxide samples (four replicates) were analyzed by INAA and direct current Arc OES (DC Arc OES). This sample (zirconium oxide) could not be analyzed by IM-NAA as the neutron flux parameters (f and α) of irradiation position at Dhruva reactor are not available. In addition to the comparison of results with relative INAA and ICP-OES, a nimonic alloy CRM—BCS CRM 310/1—was analyzed using IM-NAA as well as relative INAA methods.

2. Materials and methods

2.1. About the samples

Different alloys are important in almost all fields of modern science and technology. The elements Fe, Ni, Cr, Zr, Pb, and Ti are the base materials for most of the alloys. High nickel alloys are used in the chemical, petrochemical, turbine, and aerospace industries. High Ni alloys (having 50–80% Ni), such as nimonic alloys, are used in the pressure lining of steam generators and are also preferred over stainless steels as structural materials in fast nuclear reactors. Swelling under the prevailing irradiation conditions of high temperature is low for these alloys, and they are less prone to stress corrosion cracking.

Zirconium occurs in nature, mostly in the form of a silicate mineral known as zircon, which is extensively used in the glass and ceramic industries. Zirconium oxide (ZrO_2, zirconia), prepared from zircon, is used as traditional and advanced ceramics and as a refractory material. It is used as a material for machinery wear parts, insulations and coatings, and solid electrolytes for fuel cells. It is also used as a raw material for the preparation of various zirconium-based alloys. Zirconium forms alloys with a variety of metals, such as Nb, Ni, Fe, Sn, and Cr. These elements are added to zirconium to improve the mechanical properties and to decrease corrosion by water at high temperatures. Tin, as an alloying element, is most effective in imparting corrosion resistance without seriously affecting neutron economy in nuclear reactors [30]. Zirconium alloys are mostly used for fuel cladding and core components in water-cooled nuclear power reactors, viz. boiling water reactors, pressurized light water reactors, and pressurized heavy water reactors. Zirconium-based alloys are the major structural components for nuclear (thermal) reactors, namely,

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