Ain Shams University
Women's College for Arts,
Science and Education

"Application of Thin Layer Activation Technique for Wear and Corrosion Studies in Stainless Steel Using Neutron Sources"

Thesis submitted for
M.Sc. Degree of Science "Nuclear Physics"

By

Reem Faseil Ibrahim Mohamed

Supervised by

Prof. Dr. Zeinab Abdou Saleh
Prof. of Nuclear Physics
Experimental Nuclear Physics Department
Nuclear Research Center,
Atomic Energy Authority

(Late)Prof. Dr. Ahmed Morsy
Prof. of Radiation Physics
Physics Department
Women's College for Arts, Science
and Education, Ain Shams University

Dr. Afaf Abd El-Lateef Nada
Assistant Prof. of Nuclear Physics
Physics Department
Women's College for Arts, Science
and Education, Ain Shams University

Dr. Hassan Ibrahim Hassan
Assistant Prof. of Nuclear Physics
Cyclotron Physics Department
Nuclear Research Center
Atomic Energy Authority

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Date of Approval
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Approval of Faculty Council
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Approval of University Council
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ABSTRACT

In this work elemental analysis for three types of stainless steel samples was performed to compare between their compositions. First the stainless samples were analyzed using Energy Dispersive X-ray (EDX) Spectrometer and Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) as conventional tools for elemental analysis. Second, the samples were subjected to detailed neutron activation analysis (NAA) using Pu-Be neutron source with applying γ-rays spectroscopic measurements for the irradiated samples. The first sample was in the form of thin foils. Eight radioactive isotopes were detected in the measured spectra namely ^{56}Mn, ^{59}Fe, ^{58}Co, ^{60}Co, ^{24}Na, ^{187}W, ^{99}Mo and ^{51}Cr which resulted from different neutron reactions with this sample. The other two samples were commercial and the NAA results for one of them show that all of the elements reported in the foil sample are the same except the absence of Mo and the presence of Cr.On the other hand the third sample shows a different composition where only Mn, Fe, and Ni were identified from the measured γ- ray spectra. Stacks of irradiated stainless steal foil and pellets were measured to obtain the activity as a function of thickness using the most intense gamma ray lines of the produced radionuclides. The obtained linear activity-thickness relations for the measured radionuclides were fitted to determine the slope and the maximum thickness which can be measured by this technique. The comparison between these curves showed that the most sensitive radioisotope for detecting slight changes in the thickness is ^{51}Cr which is formed through the ^{50}Cr (n,γ) ^{51}Cr reaction.

Summary

The thesis contains four chapters in addition to this summary and a conclusion.

Chapter I: General Introduction

This chapter presents the different parameters affecting the wear measurements. The method of radioactive techniques for wear measurements is presented in this chapter. It includes also some information about the irradiation sources for studying wear and corrosion.

Chapter II: Neutron Sources and Neutron Activation Analysis (NAA)

Brief information about neutron classification and types of neutron sources are presented. Also it includes an overview about some of the neutron interactions, neutron cross-section, neutron thermalization and moderation, and shielding of neutrons. Different types of neutron activation analysis were also discussed from different point of views such as advantage & application, forms and elements applicable for NAA. Determination of the neutron flux by thin foil technique as well as the aim of the present work is also included.

Chapter III: Experimental Setup

This chapter includes the general classification of the stainless steel and the composition of each type. It also contains a description about the used samples and the other techniques used to analyze the unknown stainless steel samples qualitatively and quantitavely. A description of the experimental technique used to study the irradiated samples of stainless steel and the different parameters affecting the measurement were also discussed and presented.

Chapter IV: Results and Discussions

A brief view over on the delayed gamma ray and prompt gamma ray analyses is presented in this chapter. It also includes the experimental data for a three of unknown stainless steel samples using neutron activation technique. Energy spectra for the irradiated samples are presented. Thermal neutron flux using a thin gold foil of 10μm thickness was determined and found to be $2.7x10^3$ neutron/cm^2.sec. Mn concentration in a sample of well known composition $Ni_{0.6}$ $Mn_{0.4}$ Fe_2O_4 and weight 852mg was determined and compared with the concentration obtained from an irradiated sample, a good agreement was achieved. Elemental analysis of the three stainless steel samples was carried out. Comparison between the qualitative analysis of our results and those obtained using EDX and ICP-AES is also presented. Thin layer calibration curves for some radionuclides for enhanced corrosion and enhanced wear were constructed and included also the conclusion.

الملخـــــص

الفصل الأول: مقدمة عامة

يحتوى هذا الفصل على استعراض للعوامل المؤثرة على عمليات التآكل الناتج عن العمليات الميكانيكية أو الكيميائية. وفى هذا الفصل أيضا تم استعراض لتقنية قياس التآكل بالطرق الأشعاعية. كما تم ايضا استعراض للمصادر المشعة التى يمكن استخدامها فى هذه الدراسات.

الفصل الثانى: مصادر النيوترونات وتقنية التحليل بالتنشيط النيوترونى

١ ـ تم تصنيف وتحديد انواع المصادر النيوترونية.
٢ ـ القاء نظرة عامة عن كل من تفاعل النيوتـرونات – المقاطع المستعرضة – تهدئة النيوترونات. والدروع المستخدمة للحماية من النيوترونات.
٣ ـ التحليل عن طريق التنشيط النيوترونى . مزاياه وتطبيقاته . وكذلك اشكال ونوعية العناصر التى يمكن تحليلها بهذه الطريقة.
٤ ـ طريقة تحديد الفيض النيوترونى باستخدام تقنية الشرائح الرقيقة
٥ ـ الهدف من الدراسة.

الفصل الثالث: الأجهزة والمعدات

١ ـ يحتوى هذا الفصل على تصنيف لأنواع الصلب ومكون كل نوع من الأنواع. كما يحتوى أيضا على وصف لأنواع الصلب محل الدراسة والتقنيات الأخرى المستخدمة فى تحليل هذه العينات
٢ ـ شرح تفصيلى للتقنية المستخدمة وهى التحليل بالتنشيط النيوترونى والعوامل المؤثرة على هذه القياسات.

الفصل الرابع : النتائج والمناقشة

ويشمل هذا الفصل مقدمة مختصرة عن كل من أشعة جاما المباشرة والمتأخرة كما يشمل أيضا على:

أولا:- التحليل العناصرى بالتنشيط النيوترونى

١ - النتائج العملية لثلاث عينات من الصلب مجهولة التركيب ومعرضة لفيض من النيوترونات المنبعثة من مصدر بلوتونيوم –بريليوم.
٢ - تم تحديد الفيض النيوترونى الحرارى باستخام شريحة رقيقة من الذهب عالى النقاوة .
٣ - لتأكيد صحة النتائج تم استخدام عينة معلومة التكوين $Ni_{0.6}\ Mn_{0.4}$ Fe_2O_4 والوزن 852 جم وتم حساب تركيز المنجنيز بها ولنفس العنصرتم حساب التركيز من الطيف الجامى الذى تم الحصول عليه بعد تشعيع العينة باستخدام قيمة الفيض الذى تم الحصول عليه حيث وجد ان نسبة الخطأ لا تتعدى 1.5%
٤ - تم عرض بعض الأطياف النووية التى تم الحصول عليها باستخدام مطياف جامى عالى النقاوة ذو قوة تحليل عالية سواء كان ذلك بعد تعريضها مباشرة أو بعد اضمحلالها لفترة مناسبة وذلك لتحديد النظائر المشعة الناتجة والتخلص من أى تداخلات بين الخطوط الجامية.
وللسبب ذاته تم قياس الأطياف الجامية على مدى شهر بعد انتهاء التشعيع.
تم الحصول على العناصر المكونة لكل نوع من أنواع الصلب المستخدم ومقارنة ما تم الحصول عليه من نتائج بتلك التى تم الحصول عليها من التقنيات الأخرى.

ثانيا: تقنية التنشيط الأشعاعى للشرائح الرقيقة وأهميتها فى تحديد التآكل

تم محاكاة لعمليات التآكل التى تحدث فى بعض الأجزاء الميكانيكية التى تتعرض للأحتكاك أثناء الدوران وذلك عن طريق استخدام تقنية الشرائح المتراصة أو عن طريق التآكل الكيميائى باستخدام الماء الملكي، حيث تم رسم العلاقة بين النشاط الأشعاعى لبعض العناصر التى تكونت بعد تعريض العينات للمصدر النيوترونى كدالة فى السمك ومنها تم تحديد بعض البارامترات وتتضح أهمية هذه الطريقة فى أمكانية استخدامها فى المجالات التطبيقية المختلفة.
ويحتوى هذا الفصل أيضا على الخلاصة.

The Aim of the Work

The aim of the present work is first to study the elemental analysis of some stainless steal samples using the industrial neutron activation (NAA) technique, for the purpose of using these elements for further study concerned with thin layer activation which utilizes radionuclides to determine surface degradation processes such as corrosion and wear.

The principle of TLA is the creation of an appropriate radionuclide at a well defined depth over a selected area. This activation is achieved by charged particles accelerator or by neutrons. For this purpose calibration curves can be constructed either by mechanical or chemical etching or by using stacked foils. The activated sample is used for measurements of its residual radioactivity after removing thin layers of material by mechanical or chemical etching. The variation of the activity versus the removed thickness is then plotted. This technique has been used mainly for wear testing elated to the automotive industry applications.

Contents

CHAPTER (2)

Neutron Sources and Neutron Activation Analysis (NAA)

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RESULTS and DISCUSSIONS