Precise Determination of Br in ABS Resin by Instrumental Neutron Activation Analysis

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INTRODUCTION: National Metrology Institute of Japan is responsible for developing certified reference materials (CRMs) and for establishing the traceability of SI (The International System of Units) on chemical metrology in Japan. To establish SI traceability, the primary method of measurements should be applied to the characterization of the CRMs. Recently, neutron activation analysis (NAA) using comparator standard is recognized as a potential primary ratio method [1]. Despite the potential of NAA as primary ratio method, the evaluation of the measurement uncertainty is required in any analysis. In general, there are three main components of uncertainty in NAA, that is, sample preparation uncertainty, neutron flux homogeneity, and gamma ray measurement uncertainty. Usually, flux monitor is used to correct the neutron flux in-homogeneity. However, although the flux monitor can correct the neutron flux variation using the count rate of the known amount of the monitor nuclide, it does not reflect the neutron flux of the actual sample. The most practical method to eliminate neutron flux in-homogeneity and to improve gamma ray measurement uncertainty is an internal standard method [2]. In this paper, we presented that notable capability of internal standardization in NAA for determination of Br in acrylonitrile butadiene styrene copolymer (ABS) resin as a candidate CRM.

EXPERIMENTS: The ABS resin candidate CRM was produced by a mixing machine. The calibration solution of Br was prepared from NMIJ primary standard solutions. The Au solution for the internal standard for Br was prepared from a high purity metal. The calibration solutions contained Br and Au as internal standard. One hundred mg of the ABS resin samples was used for Br analysis. The Au solution was added to the samples before neutron irradiation. The neutron irradiations were performed by KUR (Kyoto University Research Reactor) TCP\textsuperscript{n} (thermal neutron flux: $8.0 \times 10^{10}$ cm\textsuperscript{-2}s\textsuperscript{-1}) for 6 h. The $\gamma$ ray measurement system consisted of a Canberra GC4070-7500 Ge detector and a Laboratory Equipment Corporation MCA600.

RESULTS: It was found that the $^{198}$Au sensitivity (cps/µg) varied according to the sample position in the irradiation capsule. The relative standard deviation of the $^{198}$Au sensitivity (cps/µg) was 3.8 %. The uncertainty related to the neutron flux homogeneity significantly contributes to the overall uncertainty, if an internal standard is not applied. The amount of Br in the ABS resin sample was determined by calibration curve method using internal standardization. The linearity of calibration curve was improved by internal standard. The relative standard uncertainty related to the calibration curve linearity was improved to 0.36 % to 0.87 %. The analytical results of Br in BCR-681 certified reference material of polyethylene resin by proposed method were in excellent agreement with the certified value only using internal standard.

The analytical results of Br in ABS resin sample by proposed method was $575$ mg/kg ± $8.0$ mg/kg (n=8). The relative expanded uncertainty (k=2) was 1.4 %.

REFERENCES:
CO12-2  Thickness and Density of Adsorbed Additive Layer onto Metal Surface under Rolling Friction Evaluated by Means of Neutron Reflectometry

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INTRODUCTION: Tribology is one of academic disciplines covering friction, wear and lubrication in engineering for a lot of industrial applications such as bearings, gears, engine pistons in car and so on. Formation of adsorbed additive layer on metal substrates has attracted attention as an effective way to reduce friction. There are a lot of studies investigating the relationship between the kind of additives and their tribological properties, but the formation process of the adsorbed layer is still unknown. On these backgrounds, this study investigated the formation and desorption process of adsorbed additive layer onto metal surface by means of attenuated total reflection infrared spectroscopy (ATR-IR) and neutron reflectometry (NR) with time slice mode.

EXPERIMENTS: Thin copper film was deposited onto silicon wafer as target substrate for the study. Poly-alpha-olefin and palmitic acid were selected as base oil and oiliness additive, respectively. The time dependences of IR absorbance profiles and neutron reflectivity profiles are shown in Figs. 1 and 2. We can see that the IR peaks from COO bonds gradually grown up after the injection of base oil with oiliness additive. On the other hand, the neutron reflectivity profiles did not change regardless of time. These results indicated that the physisorption of oiliness additive molecules was quickly completed and the chemisorption was gradually proceeded. To investigate the effect of rolling friction, we designed the sample holders with rolling friction mechanism for ATR-IR and NR. The obtained results are shown in Figs. 3 and 4. The IR profiles showed the desorption of additive layer, but the NR profiles did not change even after the rolling friction.

CONCLUSIONS: The additive molecules made the condensed layer around the top surface by physisorption, and they gradually reacted by chemisorption. The rolling friction process peeled the adsorbed additive molecules off from the surface temporarily, but the layer was quickly repaired. ATR-IR and NR were quite effective to see the adsorption and desorption processes with separating ‘physisorption’ and ‘chemisorption’.

Fig. 1. Time dependence of IR absorbance peaks

Fig. 2. Time dependence of neutron reflectivity profiles obtained by SOFIA at BL16 in J-PARC MLF

Fig. 3. Time dependence of IR absorbance peaks after rolling friction

Fig. 4. Time dependence of NR profiles after rolling friction
INTRODUCTION:
New hybrid detector called B-10+ detector has been developed by GE Reuter-Stokes. The detector is what combines B-10 lined proportional counter with He-3 counter. A big feature of B-10+ detector is reducing costs while the performance doesn’t decrease much. We have developed NDA system against the Fukushima debris, and then we are considering applying the B-10+ detector instead of He-3 detector that has been most popular for general NDA systems. The original experiment plan at KUR was twice experiments at the B-4 port, and once experiment with high gamma ray and neutron flux at the area under the core. We could carry out the first experiment in 2014. In the experiment using the B-4 port, we obtained fundamental data of B-10 detector such as the pulse shape and plateau.

EXPERIMENTS:
The B-10+ detector is 100cm in length. And the gas pressure including He-3 is approximately 1 atm. Three types of amplifiers were used. A current amplifier and charge amplifier are developed by our group, and another is a charge amplifier from PDT Inc. He-3 detector (4 atm) was also used in order to compare detection efficiency with B-10+ detector. The high voltage power supply was ORTEC 556H, DC supply was KIKUSUI PDM-3, and counter was ORTEC 872.

RESULTS:
As shown Fig.1, the current amplifier and charge amplifier caused pile-up, which was also observed on an oscilloscope. Therefore we measured neutron flux through an absorber, the plateau region was observed clearly. Due to the limited machine time, data of the attenuated flux was not obtained by using a current amplifier, and data after the plateau region was not done. Some measurements by He-3 were carried out. As an easy reference, measurement result by He-3 detector indicates that B-10+ detector has 40% detection efficiency of He-3 detector. Fig.2 shows a pulse from a charge amplifier on an oscilloscope. The width was approximately 1 μs that was the same degree as using He-3 at another measurement. Fig.3 shows an output spectra for a B-10+ detector. A sharp peak of He-3 was not observed. The reason is considered that the effect of the He-3 gas is not stronger than the lined B-10 since the amount of He-3 gas is reduced. We will investigate characteristics of B-10+ detector in high gamma ray field as future work. After that, we will try experiments applying B-10+ detectors to NDA system.
CO12-4 Evaluation of Multiwire-type Two-Dimensional Neutron Detector with Individual Line Readout

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INTRODUCTION: Multiwire-type two-dimensional neutron detectors using helium-3 gas are widely used in neutron scattering experiment facilities because of their high detection efficiency, low gamma-ray sensitivity, and excellent long-term stability. We have developed an advanced multiwire-type two-dimensional neutron detector system without compromising the otherwise excellent performance of the conventional detector [1, 2]. A short response time and better spatial resolution can be obtained in this system by using an individual line readout method. Additionally, optical devices have been developed and incorporated in the system for long-distance signal transmission and insulation between the detector heads and the signal processing circuits. In this present study, we performed neutron irradiation experiments at KUR.

EXPERIMENTS: The multiwire element, which is developed for individual line readout, has 128 anode wires, 128 cathode wires for the y-axis, 128 cathode strips for the x-axis, and a wire/strip pitch of 1 mm. The amplifier-shaper-discriminator boards have a total of 256 channels to process each cathode signal line. Field-programmable gate array based digital signal processing and digital signal encoding are used to calculate signal position with a sampling frequency of 106 MHz. Dedicated optical signal transmission devices are designed to use multimode optical fibers at a wavelength of 850 nm. List-mode data with the x- and y-positions and the time of flight are recorded using a data acquisition system (MPA-3, Fast Comtec). Neutron irradiation is performed using a neutron beam line CN3 at the KUR.

RESULTS: Fig. 1 shows a typical response when a 1.0 × 1.0 mm² neutron beam is irradiated on the detector, and the response at the peak position onto the x-axis is shown in Fig. 2. As seen in Fig. 2, there are some background signals. It is considered that these signals arise from neutron scattered by the beam line, the collimator, the window and the fill gas. Fig. 3 shows the reactor power dependence to the measured counts of the developed detector system. The developed detector showed a linear response up to the full power, 5 MW, of KUR reactor and the counting rate at 5 MW was approximately 2.1 x 10⁵ cps.

REFERENCES:
Development of Neutron Resonance Densitometry for Nuclear Material Quantification in Melted Fuel

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INTRODUCTION: Neutron Resonance Densitometry (NRD) [1, 2] is an assay technique of nuclear materials in particle-formed debris that contains various nuclides, such as of structural materials and the others. It is a combination of two techniques: Neutron Resonance Transmission Analysis (NRTA) [3], and Neutron Resonance Capture Analysis (NRCA) [4] or Prompt Gamma-ray Analysis (PGA). Containing nuclides in a sample are identified by NRCA/PGA. The information of NRCA/PGA is used in NRTA analysis to quantify nuclear material isotopes. A neutron time of flight (TOF) method is used in NRD measurements. A facility consisting of a neutron source, neutron flight paths, and detectors, is required. One of the distinctive features is utilization of a short flight path; this increases the neutron beam strength, and reduces construction cost in a practical applications. On the other hand, that reduces resolving power in TOF measurements, and increases neutron background. In order to investigate the effect of neutron flight length, we carried out NRTA experiments with a short neutron flight path constructed at the Kyoto University Research Reactor Institute - Linear Accelerator (KURRI-LINAC).

EXPERIMENTS: Experiments were carried out with a 7.5-m flight path. The experimental room and the neutron source were separated by 2-m-thick concrete wall. A 2-m-long neutron transport tube was installed in the experimental room as shown in Fig. 1. The center of it was an Al vacuum chamber, the cavity of which was 200 mm in diameter; both sides were shielded by 2-mm-thick Al plates. The chamber was surrounded by 5-mm-thick B4C-doped silicone rubber (B4C 40%). The outside of the rubber was high density polyethylene plates (460 x 460 mm). The neutron beam was collimated to be 50 mm in diameter in front of the transport tube. A 6Li scintillation detector (Saint-Gobain GS20; 100 x 100 x 10 mm) was used to measure transmission neutrons. The incident neutron flux was monitored by a BF3 neutron detector placed near the exit of the concrete wall.

Sample and filter plates (100 x 100 mm) were placed in front of the transport tube: In (0.2 mm), Ag (0.25 mm), W (0.5 mm), and Pb (10 mm). The neutron pulse width was controlled by the pulse width of the LINAC electron beams, which were 500 ns, 2 μs and 4 μs.

RESULTS: Figure 2 shows TOF neutron transmission spectra. The ordinates are the normalized neutron counts, and the abscissa time interval after neutron pulse generations. The dips in the spectra around 192 and 102 ns are resonances of 183W at 7.64 eV and 27.0 eV, respectively. Since the origin of the spectra is relevant to the beginning of electron pulse injection, the ends of the dips come later as the electron pulse width increases. The dip at 27.0 eV is not clearly seen with 4-μs electron pulse width.

SUMMARY: In measurements of NRD, the quantity of the nuclear materials is determined from NRTA spectra in the neutron energy range of less than 30 eV [1, 2]. At KURRI-LINAC, we have examined NRTA with a 7-m flight path. The resonance dips of 183W at 27 eV was successfully observed with an electron pulse width less than 2 μs. This indicates the applicability of short-flight-path TOF system to nuclear material quantification. The importance of the pulse width of neutron beam should be noted. Both simulation studies and experiments are expected for realistic design of a TOF facility of nuclear material quantification.

REFERENCES:  
INTRODUCTION: DeeMe [1] pursue mu-e conversion in nuclear field, which will be a clear evidence of the new physics if it is found, because Standard Model of elementary particle physics predicts it beyond an experimental reach. Detector is required to measure electron momentum precisely after a huge pulse of prompt timing charged particles, whose width and instantaneous hit rate is expected to be approximately 200ns and 70GHz/mm². Such a large input will make usual wire chamber detectors inactive due to space charge effect. We developed a new technique to make a chamber to be tolerant to a large input pulse.

EXPERIMENTS: Experimental setup is shown schematically in Fig. 1. Electron beam collimated to 16mm x 19mm penetrates Multi-Wire Proportional Chamber (MWPC) and beam counters. Beam rate is tuned changing current of electron gun heater. A huge pulse of prompt timing is emulated by tuned pulse from electron beam gun. Delayed electron signals are emulated by field emission electrons. Electrical field contour inside the chamber calculated is shown in left of Fig. 2. Anode wires with 1450V and potential wires with 0V are put alternately. During a huge pulse are coming, potential wires are applied with 1450V as shown in right of Fig.2. With this setting, slope of electric field near anode wire become gentle that avalanche multiplication is suppressed. Temporary muting the chamber during a burst pulse is expected to help to suppress the space charge effect. Rectangular pulsed high voltage is applied to potential wire to recover quickly after the blinded period. Because of the very high and rapid voltage up and down motion, readout electronics suffers from large current input derived from the HV switching. We also developed a readout system which tolerates to high current input.

RESULTS: Fig. 3 shows an output result of the chamber. Electrons from beam gun tuned to be 70GHz/mm² in instantaneous hit rate are not amplified in the chamber with applying pulsed high voltage to potential wire. This enabled single electron detection coming after the burst pulse. Noise derived from the HV switching is always the same, so we can eliminate the noise by subtracting averaged waveform of plural numbers of events. We succeeded to obtain a clear electron signal after the burst pulse avoiding the space charge effect by a burst pulse of charged particles.

REFERENCES:
Aside the pros and cons of the use of nuclear power as an energy source, it seems to be useful in understanding the nuclear reactors to describe them as an ethnography based on the narrative data of nuclear researchers. By describing the reactor as a scientific ethnography, it is expected to be highlighted the problem of nuclear reactors and low-level radiation environment from a new angle. Then, it is expected to provide further knowledge for living in the contact zone between the field we live in safely (normal) and low-level radiation field (pathological), and going into patho and/or coexisting with patho.

This is a very unique study that an ethnographer who is a radiation physicist, a physician and also an anthropologist participates in wide-range observation of nuclear research. The researchers have been forced to include Fukushima as an enlarged research field, and it is regarded as a chance to write up the excellent scientific ethnography by doing this rather. On 2014-2015, we have mainly investigated previous research, and preliminary investigation has been carried out with biologists and physicists involved in nuclear science at the Kyoto University. Their narrative indicated here was traumatic narrative of Fukushima that suddenly appeared in their research field of nuclear science. Sometime it was a narrative of the confusion of the researchers themselves, although almost of the nuclear scientists have investigated sincerely with the nuclear reactor research as an energy source of the human being. The radioactivity that has been discarded and released has been completely isolated in a managed environment from the living space. Conversely, by this act, understanding of nuclear reactors and low-level radiation environment, and also constructing networks among various sub-specialists structurally become difficult.

The reason why researcher's confusion about Fukushima accident is so strong, I think because our wisdom as the central pillar of our ego might be extremely imbalanced state. As being symbolically shown in Fukushima, because wisdom of nuclear science has extremely hypertrophied self-righteously, once it fall into a little collapse, lack of resilience of human wisdom for stabilization and repair becomes embody.

By analyzing the narrative concerning the reactor of nuclear researchers, to describe the ethnography of nuclear reactor and nuclear waste that is also a study object of researchers themselves seems to lead to development of a new approach to the methodology of cultural anthropology. Such ethnography seems to be very useful for human society by targeting to nuclear reactor as thing. Many nuclear physicists have focused on the following statement of Prof. Wolfgang Weiss;

"The most serious damage caused by Fukushima accident is that people lost trust for science and scientists. This certainly will influence long-term human history, and we cannot recover such situation so easily. We scientists are responsible to such situation."

Latour has pointed out that the dynamic and contingent elements intervene in the process of establishing the level of intellectual authority and knowledge that circulates within the scientific community. Case of Fukushima accident shows this extremely and plainly. It is expected to also lead to scientific achievements, such as actor network theory by scientific anthropological comparison analysis of the various disciplines of nuclear reactor researchers as different cultures. And also, ethnographic research might be very helpful tool for well public understanding of reactor and/or low dose radiation field, and then might regain confidence of scientists.

The understanding and knowledge of the researchers of KUR for their acceptance of this ambitious study is greatly appreciated. A new division for Ethnographic Study for Nuclear Science is expected to be established in KUR. This research is supported by Grant-in-Aid for Scientific Research for 2014-2016, No.26580152, JSPS. We are now promoting our fieldwork and are planning to publish ethnography of nuclear reactor on 2017.

REFERENCES:
CO12-8  Effective Measures on Safety, Security, Hygiene and Disaster Prevention in Laboratories

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INTRODUCTION:
Important research aspects can be found in the following keywords such as safety, security, hygiene and disaster prevention. Nuclear research reactor is one of representative facilities together with these keywords under their operation. It is effective to investigate the latest status on practical measures on these keywords in various facilities including nuclear research reactors, to compare each other among facilities, and to discuss more effective or optimized ones for our positive safety management. Through this process, it is also effective to investigate the latest international and/or national regulations and the movement of revision of them. This total discussion contents and its fruit are directly useful for all relating laboratories.

RESEARCH APPROACH:
General research approach is as follows.
- Measures of safety management during operation or standstill status of the real facilities would be investigated. This information would be used for our research discussion on the positive and more optimized safety management.
- It would not be a single year research, but maybe two to three years research for one theme.
- Information source of facilities would not be only KUR, KUCA or the other facilities in Kyoto University, but also the Kinki university research nuclear reactor or the facility of National Institute of Fusion Science, etc. This research is an active joint-research with these relating facilities and positive researchers on safety management.
- One of the distinctive features of this research is to involve office staffs as cooperators as well as researchers and technical staffs. In The University of Tokyo, most of the members in Division for Environment, Health and Safety are office staffs who knows real situation of safety management in laboratories very well.

Discussion target in BY of 2014 was determined as “safety control in use of small amount nuclear materials” through our internal member discussion. We investigated the related regulations and their application as well as the fact-finding in use of small amount of uranium and thorium in Japanese laboratories.

USE & MANAGE OF SMALL AMOUNT of U & Th:
When using small amount of uranium or thorium up to 300g (DU, NU) and/or 900g (Th), most of safety control procedures requested as radioactive materials are exempted by related regulations. Weighing control of nuclear elements is only requested by regulation. This causes easy usage of uranium and thorium in laboratories. For example, it is convenient that uranium acetate or uranium nitrate could be used for biology research to observe chromosomes clearly. Natural occurring radioactive materials (NORM) such as minerals and seawater samples including uranium and thorium would also be troublesome sources which could be enriched through some research processes, and then would change into high concentration radioactive materials as a result legally. This is a representative technologically enhanced NORM (TENORM). TENORM is one of headache materials in laboratory. Even the amount is quite small, the nuance of terms of “nuclear” “uranium” and “thorium” will give a big impact on society when some accidents or troubles happen. We can use these materials easily and conveniently, however these are strictly controlled by the international regulations on their transfer. The latest guideline on NORM and TENORM safety management by the Japanese regulatory body was published in 2009 to support the safety management. These situations must be identified and understood by all stakeholders. Framework of related education for safety managers and users should be adequately established and applied. All stakeholders should understand their risk level and features of nuclear materials as radioactive materials. This could lead reasonable and optimized safety management in laboratories.

NEXT RESEARCH TARGETS:
Education framework, curriculum and textbooks for safety managers and users as well as relating officers to fill up the nuclear material controlling sheet for the use of small amount of uranium and thorium should be discussed. This discussion should be based on the additional investigation and analysis of the way to use, control and storage the materials in laboratories.

REFERENCES:
Development of New NiMo Neutron Mirror for Fundamental Physics

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INTRODUCTION: A large amount of CP violation (CPV) is necessary to solve matter and anti-matter asymmetry problem in the universe. However, the amount of CPV standard model predicts is too insufficient to explain it. The fact indicates that the new physics beyond standard model (BSM) exists and supplies the lack of CPV. The neutron intrinsic electric dipole moment (nEDM) is sensitive to CPV that BSM provides. Therefore, we can effectively search BSM with precise nEDM measurement.

The nEDM can be obtained from the ultracold neutron (UCN) spin precession in a storage container under homogeneous electric and magnetic fields. In the experiment, the loss of UCNs and their polarization occur due to interaction between UCNs and wall material, which make statistical precision worse. It is therefore important to develop new wall material with low loss probabilities. On the other hand, in the case that UCNs are transported from a pulsed UCN source, by decelerating fast UCNs and/or by accelerating slow UCNs properly, these UCNs can be focused on a storage container at the same time, which achieves drastic improvement of statistical precision [1]. However, if diffuse scattering induced by rough wall arises, the degree of improvement decreases. It is hence also important to develop a wall material with smooth surfaces as neutron mirrors have.

Ni-Mo FABRICATION WITH VACUUM EVAPORATED MACHINE: Nickel-Molibdenum alloy (NiMo) of 15 weight percent and 9.1 g/cm³ is known as one of the promising low loss materials. We fabricated test samples made of NiMo with KURRI vacuum evaporated apparatus and aimed for 100 nm thickness on SUS304 and Si-wafer substrates.

RESULT AND CONSIDERATION: We succeeded in NiMo film deposition itself. However, two undesirable features appeared as follows:

(1) The densities of test samples were only 6.3 to 6.6 g/cm³ by measuring XRR, which is far less than expected value, 9.1 g/cm³. Hence, our deposited NiMo film cannot be used as a wall material for nEDM.
(2) In this fabrication, a bumping phenomenon could not be suppressed. Consequently, these surfaces were so rough that it could not be used as neutron mirrors.

The facts of (1) and (2) help us to understand what phenomenon occurred while NiMo ingot evaporating. These are as follows:

(a) There is a possibility that NiMo ingot material included gasses and it caused bumping phenomenon of (2). If it true, gasses could also affect the density decrease of (1).
(b) The deposited NiMo films preserve the nature of Ni-Mo alloy; Otherwise, the density of test samples should close to the one expected for Ni, 8.9 g/cm³, because the Ni vapor pressure is 10^5 times higher than Mo at 2000 K.
(c) In spite of (b), the vapor pressure of NiMo alloy was quite low. Probably, NiMo vapor was not efficiently transported at substrates from evaporation source position, which could cause the density decrease of (1).
(d) There is a possibility that the crystal structure of the deposited NiMo films varied from that of NiMo ingot. If it true, it could affect the density decrease of (1).

(a), (b), (c) and (d) give us quite an important information for next desirable fabrication test with vacuum evaporation deposition. First, we should use degassed NiMo ingot to solve the problem of (a). Second, to compensate the shortage of NiMo vapor pressure ((b) and (c)), a next vacuum evaporated deposition should be conducted by attracting ionized NiMo vapor to substrates with a bias voltage. Third, to avoid structure change of (d), Ni and Mo are evaporated simultaneously so that the composition of deposited NiMo film becomes 15 weight percent and 9.1 g/cm³.

SUMMARY: The material with low loss probabilities and smooth surfaces is desired for nEDM experiment. NiMo of 15 weight percent and 9.1 g/cm³ is known as one of the promising low loss materials. We therefore fabricated test samples using NiMo ingot with KURRI vacuum evaporated apparatus. Although we succeeded in NiMo film deposition itself, the densities of test samples were only 6.3 to 6.6 g/cm³, and these surfaces were too rough to use neutron mirrors. To solve these problems, it is desirable that next fabrication with vacuum evaporation deposition should be performed using two evaporation source of degassed Ni and Mo ingot, and by attracting ionized NiMo vapor to substrates with a bias voltage.

REFERENCES: