# Analysis of on a Large Metallic Inclusion in the Irradiated Fuel using EPMA

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#### 1. Introduction

Metallic precipitates in the irradiated fuels affect the fuel's electric and thermal conductivities, and a large quantity of them changes the fuel performance, especially for high burn-up fuels [1].

Therefore, characterizing the metallic precipitates in a post irradiation examination is very important [2-3]. In this paper, a characterization procedure and a manner for improving the measurement accuracy by EPMA (Electron probe Micro Analyzer) were established with a well informed simulated fuel which was applied to characterizing the fission products and metallic precipitates of the simulated fuel irradiated at HANARO

## 2. Specimen preparation and Results

### 2.1 Specimen preparation

To characterize the concentration of the metal precipitates from the irradiated fuel, the radiation activity of a specimen is required to be below  $3.7 \times 10^{10}$ Bg for protecting the operator and damage to the EPMA. Although the specimen size has to be minimized, its volume could be managed by the manipulator in a hot cell. After cutting the specimen to a manageable size by the manipulator, the specimen was hot mounted with conducting resin at 150  $^{\circ}$ C and 0.6 Mpa. Too thin a specimen is liable to be broken during the hot mount, so its thickness was cut to be about 5 mm at first. And finally, a specimen of 2.5 x 5 x  $1.5 \text{ mm}^3$ (WxLxD) was fabricated by repeating the mounting and cutting several times for decreasing the radiation with a manipulator. After polishing the specimen, it was etched by an immersion method.

# 2.2. EPMA

EPMA (Electron Probe Micro-Analyzer, SX-50R, CAMECA, Paris, France) used in this experiment can treat a irradiated nuclear fuel by a special shielding of the specimen holder and is specifically shielded with lead and tungsten to permit the analysis of an irradiated

nuclear fuel. The maximum radiation activity in this EPMA is allowed to be below to  $3.7 \times 10^{10}$ Bq. The condition of EPMA was 20 kV of an electron acceleration potential and 20 nA of a beam current.

## 2.3 Metallic Precipitates of an irradiated fuel

Generally the size of a metallic precipitate is 1-2 m. A large metallic inclusion was observed during the investigation of the irradiated simulated pellet. Fig 1.a shows a photograph of a typical large precipitate observed in the specimen at  $r/r_0=0.3$ , where this precipitate has higher concentrations of palladium and ruthenium than the usual metallic precipitates, and it is accompanied by a barium oxide phase in the adjacent region. There happens to be a grain wetting when the precipitates are melted during an irradiation but this was not found in Fig. 1. Also it is not evident that a laminar structure was formed when several metals were melted during the irradiation process. Fig. 1 shows the SEM photographs for the circled region of Fig. 1 and the characteristic X-ray photographs. In the bulk, a metallic inclusion shows a higher concentration of palladium and molybdenum than the usual precipitates[4-5]. From Fig. 1, it is observed that a large metallic inclusion did not form a spherical shape. This abnormal shape might be due to a temperature distribution in the pellet during an irradiation. Fig. 2 shows the weight percent for the quantitative line in Fig. 1. In the outer region of the inclusion, Pd and Ni are dominant, while in the center region, Mo and Ru are dominant. It is also noted that Fe, Ni, Cr were found in small amounts, which were initially introduced during the milling process of the simulated fuel. But the quantity of these impure isotopes such as Fe, Ni, Cr is not insignificant. It is also understood that impurities have different phases during an irradiation and that metallic precipitates accumulate around these impurities. However, more investigations should be undertaken not only by a detailed postirradiation test but also by using various kinds of specimen fuels.

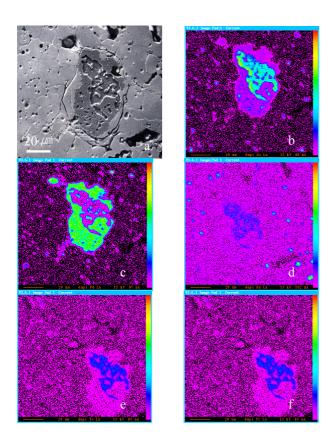


Fig.1. Secondary image photomicrograph and Characteristic X-ray photographs ( $r/r_0=0.3$ ):

(a) microstructure, (b) Mo L  $\alpha$ , (c) Pd L  $\alpha$ . (d) Ru L  $\alpha$ . (e) Tc L  $\alpha$ . (f) Rh L  $\alpha$ .

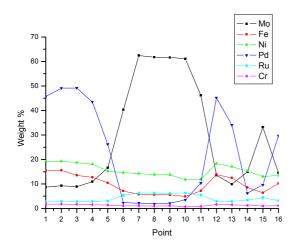


Fig.2. Weight percentage of various elements on the Fig 1.a (quantitative line)

### 3. Conclusion

A detailed analysis was performed for the metallic precipitates of an irradiated simulated fuel and the results were compared with those of a fresh simulated fuel. From the results, it was suggested that the metallic precipitate was formed during the fabrication process of the simulated fuel because the irradiation period was not long enough in the HANRO test reactor. The oxidation and reduction processes had an effect on the formation of the metallic precipitates. Also a large metallic inclusion was found and some quantitative results were provided including the weight percentage for some major elements. To establish more reliable results for a metallic precipitate and a large metallic inclusion in an irradiated simulated fuel, more post-irradiation tests should be performed with various kinds of fuels. It is believed that the approach of this study will be helpful in analyzing the performance of impurities in an irradiated fuel.

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