Electron probe microanalysis of an un-irradiated simulated fuel heated at 750℃ for SFR

Y. S. Choo*, Y. J. Kim, B. O. Yoo, S. J. Baik, Y. G. Jin, K.S. Heo, H. M. Kim, S. B. Ahn

Irradiated Materials Examination Facility, Korea Atomic Energy Research Institute,
P.O. Box 150, Yuseong, Korea, 305-600
Corresponding author: yschoo@kaeri.re.kr

1. Introduction

The electron probe micro-analyzer (EPMA) is widely applied to analyze the chemical compositions of unknown materials, especially for irradiated nuclear fuels[1].

To study and investigate on FCCI (Fuel-Cladding Chemical Interaction) which is the interaction behavior between Lanthanide element (La, Ce) and ferritic-martensitic steel of the SFR (Sodium-cooled Fast Reactor)'s irradiated simulated fuel heated at 750～800℃, which are composed of the Mitch metal, a tube, upper and lower rods[2].

Therefore, the feasibility study on SFR's irradiated simulated fuel heated at 750～800℃ was reviewed by performing two un-irradiated simulated fuels heated at 750℃ and 800℃ from transferring, loading and unloading the specimen to EPMA examination at hot laboratory in IMEF (Irradiated Materials Examination Facility)[3].

In this paper, the EPMA examination including SE (Secondary Electron) and BS (Back Scattering) images, qualitative analyses, and mapping analyses was described respectively.

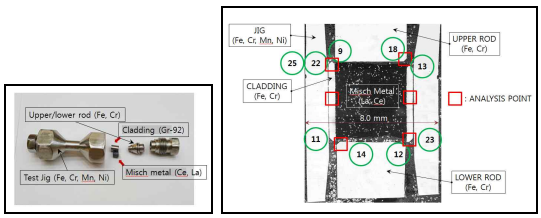
2. Experimental & Results

2.1 Specimen

To heat at 750℃ the specimen, the test jig was manufactured and assembled by master-slave manipulators at M1 hot cell as shown in Fig. 1 (a). After heated in the furnace and cooled on the working table installed at M1 hot cell. To take the macro-image of the sample for EPMA as shown in Fig. 1 (b), the test jig was cut to axial direction by the diamond saw, mounted by the hot mounting machine, and grinded the by grinding machine with grit No. #400 and #600 respectively, and polished by the polishing machine with 6u and 1u diamond paste respectively.

2.2 Electron probe micro-analyzer (EPMA)

EPMA was carried out using a state of the art shielded JEOL JXA-8230 model specially shielded and modified to permit the analysis of irradiated nuclear fuels shown in Fig. 2. This equipment has 4 WDXs (Wave Dispersive Spectrometers) to analyze elements from Boron to Uranium, and an additional function of SEM (Scanning Electron Microscope). The optimum condition of HT (kV) and beam current (A) for SEM were 20 kV and 1.0 nA, for EPMA were 20 kV and 20.0 nA respectively.



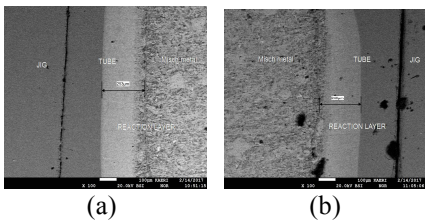
(a) (b)

Fig. 1. Specimen (a) with the Test jig before heating at 750℃, (b) Sectional image of the manufactured sample heated at 750℃ for EPMA.

Fig. 2. The appearance of the EPMA at hot lab. in IMEF.

2.3 SE and BS images of the sample

From SE and BS images of the specimen taken as shown in Fig. 3, the reaction layers between Misch metal and ferritic-martensitic steel were observed and the thickness of it would be measured.



(a) (b)

Fig. 3. SE/BS images of reaction layers for the specimen, (a) location between position No. 9 and 11, (b) location between position No. 13 and 12.

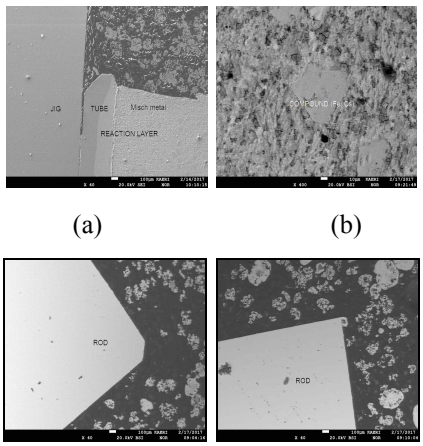
2.4 Qualitative analysis

To analyze the constituents of the specimen composed of the Misch metal, a cladding, upper and lower rods as

shown in Fig. 4, the qualitative analysis was performed under the following conditions.

- HT (kV) & beam current (A) : 20/2E-08
- CH-1 ~ CH-4 : PETJ/TAP/LIFH/LDE2

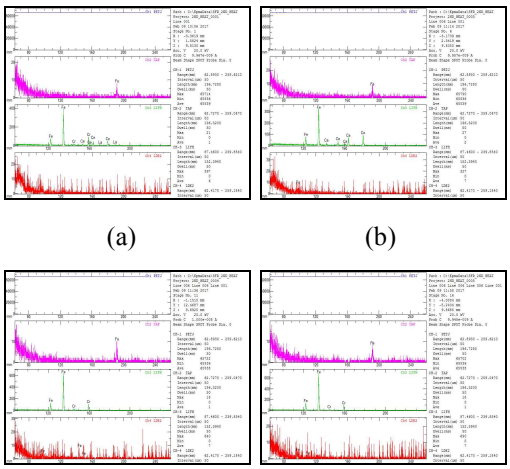
The results of qualitative analysis were shown in Fig. 5.



(a) (b)

(c) (d)

Fig. 4. Positions of qualitative analysis for (a) Misch metal and (b) compound, (c) position No. 14, and (d) position No. 18.



(a) (b)

(c) (d)

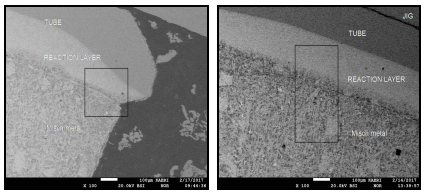
Fig. 5. Results of qualitative analysis for (a) Misch metal and (b) compound, (c) position No. 14, and (d) position No. 18.

2.5 Mapping

To observe the distribution of the constituents of the specimen composed of the Misch metal, a cladding, upper and lower rods as shown in Fig. 6, the mapping analysis was performed under the following conditions.

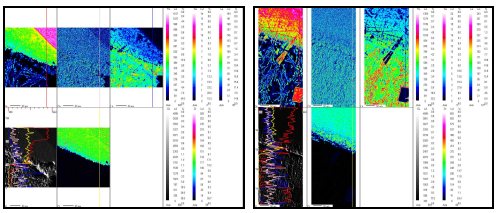
- HT (kV) & beam current (A) : 20/2E-08
- Selected element : Fe (LIF, CH-1), Cr (LIF, CH-1), Ce (TAP, CH-3), La (LIFH, CH-3)
- Pixel : 256 (1.0 um) x 256/512 (1.0 um)
- Dwell time (msec) : 200

The results of mapping analysis were shown in Fig. 7.



(a) (b)

Fig. 6. Positions of mapping analysis for (a) position No. 22 and (b) position No. 25.



(a) (b)

Fig. 7. Results of mapping analysis for (a) position No. 22 and (b) position No. 25.

3. Conclusions

Throughout the pre-test of the un-irradiated simulated fuel heated at 750℃, especially the EPMA as well as SEM examination was satisfied with good results. In particular, no reaction layer shows between the Misch metal and upper/lower rods as seen in Fig. 4 (c) and Fig. 4 (d), it says that there is no contact between two different materials to observe the FCCI during heating at 750℃. From above results and evaluations, the summary is concluded as follows;

(1) Before heating up the irradiated simulated fuel, the assembled test jig has to be tested by X-ray radiography system to confirm no gaps between the fuel and upper/lower rods.

(2) The EPMA test condition of the HT (kV) and beam current (A) values should be changed and/or increased due to strong radiation from the irradiated fuel in order to have much counts, i.e. the characteristics X-ray intensity (counts/uA).

(3) It suggests that the more accurate advanced quantitative analysis for irradiated nuclear fuel by EPMA test should be invented and developed, if possible.

REFERENCES

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[3] Y.S Choo et al., “The Development of Dismantling Machine for Capsule and HANARO fuel bundle Irradiated at HANARO”, KAERI/TR-1078/98, KAERI (1998).