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APPENDICES

Appendix A XPS Data

A1 XPS Data of Oxide on SG3 at -15° in Run 1

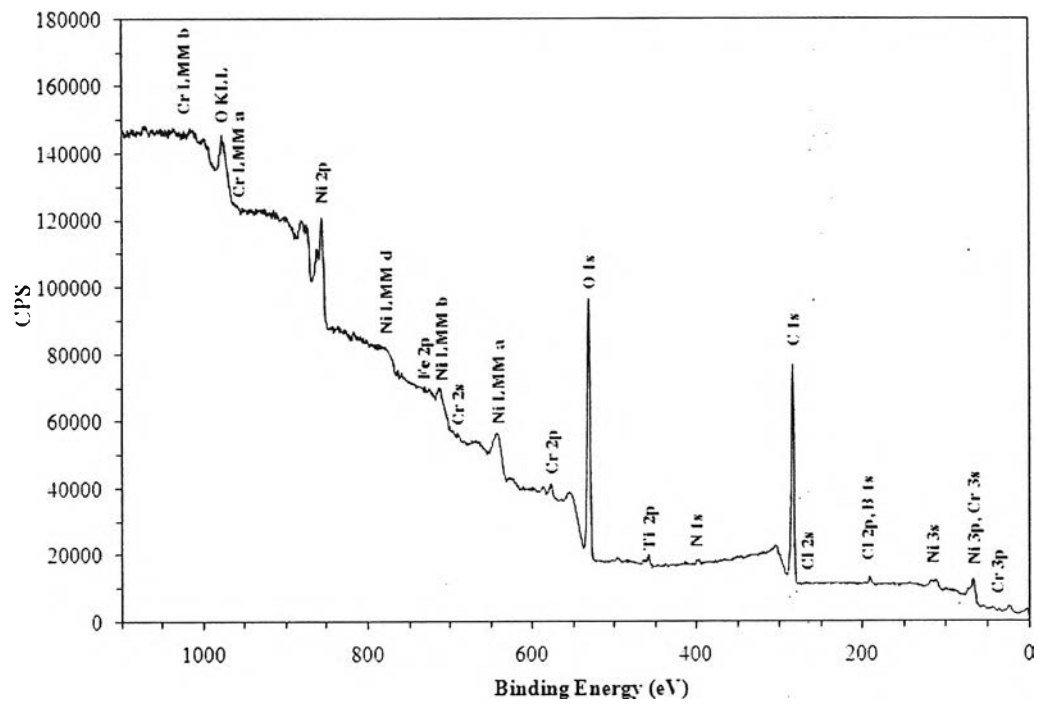


Figure A.1 XPS survey spectrum of SG3 in Run 1.

Table A.1 XPS survey signals of SG3 in Run 1

Components	Binding Energy (eV)	FWHM*	%At
B 1s	191.25	2.52	2.8
Cl 2p	197.55	2.92	0.1
C 1s	283.65	3.31	61.8
N 1s	397.05	3.86	1.1
Ti 2p	457.95	3.17	0.7
O 1s	530.75	3.81	27.5
Cr 2p	576.95	3.77	0.6
Fe 2p _{1/2}	724.65	3.11	0.3
Ni 2p _{3/2}	854.85	5.42	5.1

*Full Width at Half-Maximum

Table A.2 Ni 2p_{3/2} core-level signals of SG3 in Run 1

Components	Binding Energy (eV)	FWHM	%Peak Area of Total
Ni Metal	852.32	0.94	6.7
	855.97	2.70	0.5
	858.35	2.70	1.0
NiO	853.90	1.02	1.8
	855.61	3.25	5.6
	861.05	3.76	4.3
	864.15	2.04	0.5
	866.53	2.44	0.5
Ni(OH) ₂	855.08	1.16	5.9
	855.85	2.29	35.8
	857.87	1.59	2.4
	860.66	1.06	1.1
	861.66	4.65	31.1

Table A.2 Ni 2p_{3/2} core-level signals of SG3 in Run 1 (Cont.)

Components	Binding Energy (eV)	FWHM*	%At
Ni(OH) ₂	866.62	3.01	2.9

From Table A.2	NiO	13%
	Ni(OH) ₂	79%
	Ni Metal	8%

Table A.3 Cr 2p_{3/2} core-level signals of SG3 in Run 1

Components	Binding Energy (eV)	FWHM	%Peak Area of Total
Cr ₂ O ₃	575.70	1.10	27.2
	576.71	1.10	26.3
	577.49	1.10	14.1
	578.49	1.10	6.0
	579.30	1.10	3.8
Cr(OH) ₃	577.30	2.50	22.6

From Table A.3	Cr ₂ O ₃	77%
	Cr(OH) ₃	23%

Table A.4 Ti 2p core-level signals of SG3 in Run 1

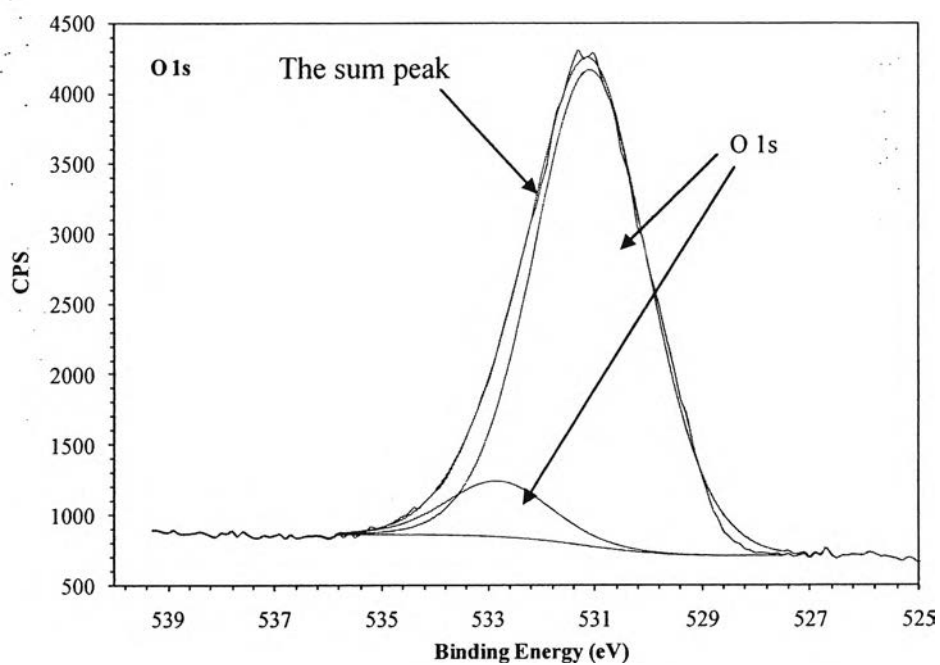
Components	Binding Energy (eV)	FWHM	%Peak Area of Total
Ti 2p 3/2 Ti(IV)	458.30	1.80	100
Ti 2p 1/2 Ti(IV)	464.02	2.30	0

Table A.5 O 1s core-level signals of SG3 in Run 1

Components	Binding Energy (eV)	FWHM	%Peak Area of Toatal
O 1s	531.07	2.52	90.7
O 1s	532.80	2.25	9.3

Table A.6 C 1s core-level signals of SG3 in Run 1

Components	Binding Energy (eV)	FWHM	%Peak Area of Total
C-C, C-H	284.80	1.37	84.0
C-OH, C-O-C	286.30	1.37	11.1
C=O	287.70	1.37	2.4
O-C=O	288.98	1.37	2.5

**Figure A.2** O 1s core level spectra of SG3 in Run 1.

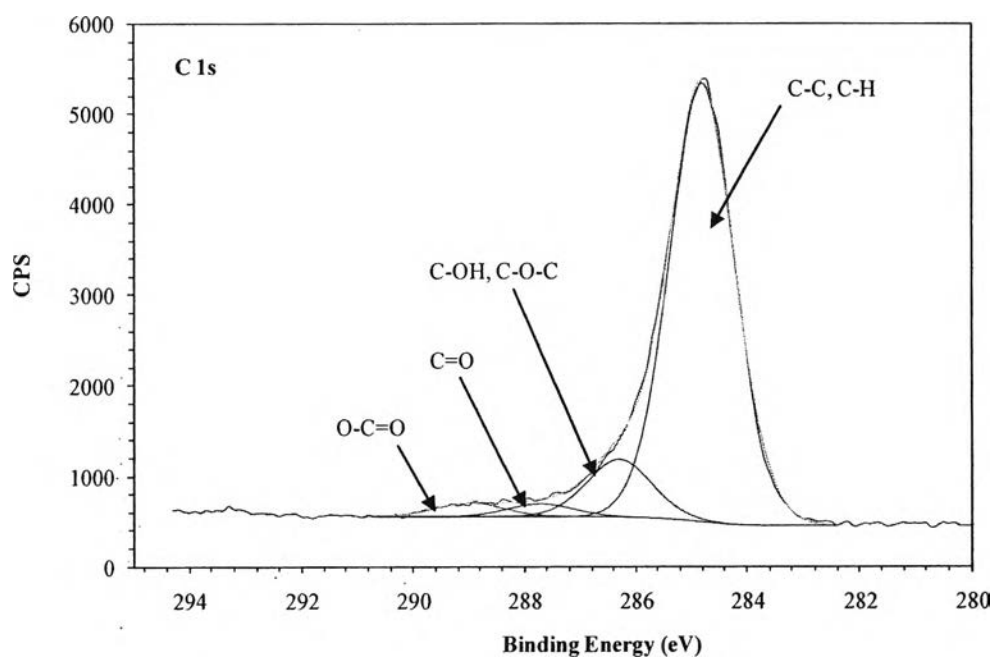


Figure A.3 C 1s core level spectra of SG3 in Run 1.

A2 XPS Data of Oxide on SS304 in Run 3

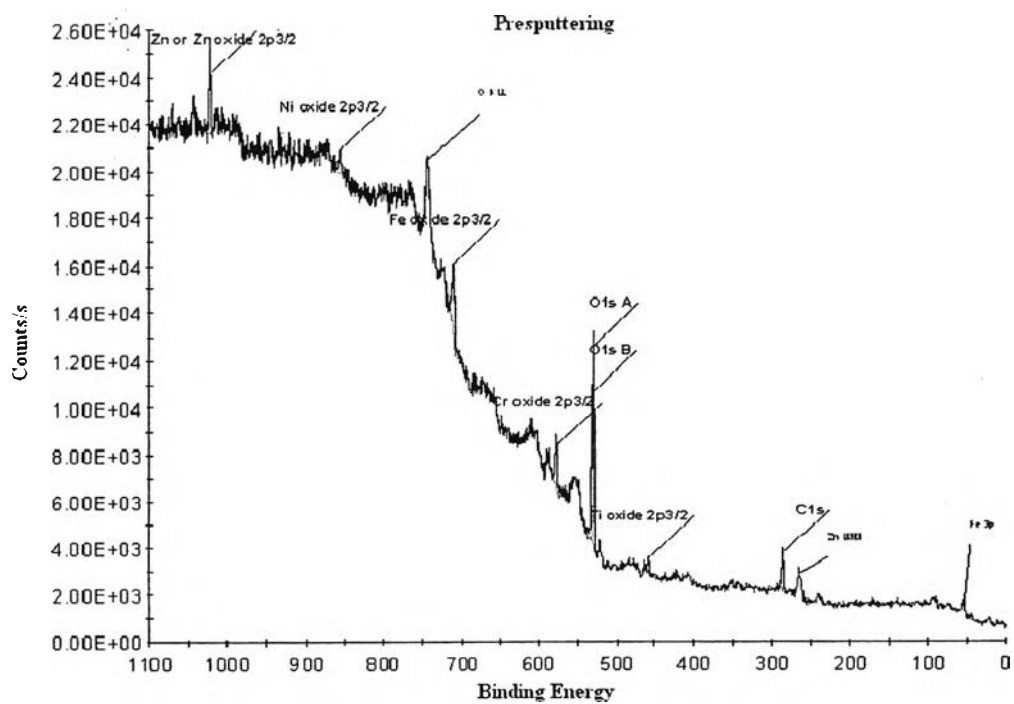
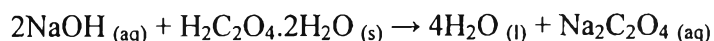


Figure A.4 XPS survey spectra of SS304 before sputtering in Run 3.

Appendix B Coolant Preparation

B1 Standardization of NaOH Solution

The molarity (moles per liter) of the NaOH solution can be determined by titration with oxalic acid solution, the primary standard solution; i.e., measuring the volume of NaOH solution that reacts with a known weight of oxalic acid, $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$. Since oxalic acid contains two replaceable hydrogens it will require two OH^- to remove them; or, it takes two moles of sodium hydroxide to neutralize one mole of oxalic acid as following equation:



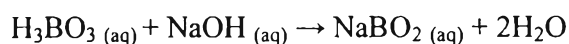
Ten ml of 0.2315 M oxalic acid solution was pipetted into a 250-ml Erlenmeyer flask. Three drops of phenolphthalein were added into the flask and the solution was swirled well. The oxalic acid solution was titrated with the sodium hydroxide solution and swirled during the titration. The NaOH solution was added continuously until the mixture turned a permanent pale pink color. At this point, additions of NaOH solution were stopped and the volume of NaOH solution used was recorded. In a sample, two titrations were required. Here is the example of calculation for NaOH concentration.

Concentration of oxalic acid	0.2315 M
Volume of NaOH used	20.525 ml
Volume of oxalic acid sample solution	10 ml

Hence, concentration of NaOH in the solution = $2 \times 0.2315 \times 10 / 20.525 = 0.2256 \text{ M}$

B2 Boron Concentration Determination

Boron concentration from boric acid can be determined by titration with NaOH solution (Irvine, 1970; EPRI NP-7077, 1990). Boric acid alone cannot be titrated to a precise volumetric endpoint because it is a weak acid. Polyhydroxyl organic compounds are used to form a complex acid with boric acid (as strong acid) that can then be titrated with NaOH solution. In this work, mannitol was used as the polyhydroxyl organic compound. The reaction between boric acid and sodium hydroxide is shown as follows:



Ten ml of boric acid was pipetted into a 250-ml Erlenmeyer flask. Three grams of mannitol were added, together with 10 ml of de-ionized water and three drops of phenolphthalein. The solution was swirled well. Then the boric acid solution was titrated with the sodium hydroxide solution (0.2256 M) and swirled during the titration. Additions of the NaOH solution were continued until the boric acid solution turned a permanent pale pink color. At this point, additions of NaOH solution were stopped and the volume of NaOH solution used was recorded. In a sample, two titrations were required. Here is the example of calculation for boron concentration.

Concentration of NaOH	0.2256	M
Volume of NaOH used	20.85	ml
Volume of boric acid sample solution	10	ml

Hence, concentration of boric acid in the solution = $0.2256 \times 20.85 / 10 = 0.4704 \text{ M}$

Molecular weights of boric acid and boron are 61.8 and 10.8, respectively. Therefore, 0.4704 M of boric acid = $0.4704 \times 61.8 \times 10.8 \times 1000 / 61.8 = 5080.32 \text{ ppm B}$

B3 Preparation of Coolant Containing Boron and Lithium

In this study, the pH_T of coolant is between 6.9-7.4 and is adjusted by using 2000 ppm B from boric acid solution and 4.89 ppm Li from lithium hydroxide solid ($\text{LiOH}\cdot\text{H}_2\text{O}$). Boric acid (5080 ppm B) solution was pipetted for 394 ml into 1000-ml volumetric flask. Lithium hydroxide solid 29.4805 mg was dissolved in de-ionized water and transferred into the same 1000-ml volumetric flask. Then, de-ionized water was added into the volumetric flask until the volume of solution was 1000 ml. The solution pH was measured with a pH meter and the boron concentration determined by titration with 0.2256 M NaOH solution. The volume of 5080 ppm B solution used to prepare 2000 ppm B solution and the amount of lithium hydroxide solid used to prepare 4.89 ppm Li can be determined as follows:

Determination of the volume of 5080 ppm B solution used

$$C_1 V_1 = C_2 V_2$$

$$(5080 \text{ ppm B}) \times \text{Volume of 5080 ppm B used} = (2000 \text{ ppm B}) \times (1000 \text{ ml})$$

$$\text{Volume of 5080 ppm B} = (2000 \text{ ppm B}) \times (1000 \text{ ml}) / (5080 \text{ ppm B})$$

$$\text{Volume of 5080 ppm B} = 394 \text{ ml}$$

Determination of the amount of lithium hydroxide solid used

Molecular weights of $\text{LiOH}\cdot\text{H}_2\text{O}$ and Li are 41.96 and 6.96, respectively.

$$\text{Therefore, the amount of } \text{LiOH}\cdot\text{H}_2\text{O} \text{ solid used} = 41.96 \times 4.89 / 6.96 = 29.4805 \text{ mg}$$

B4 Preparation of Coolant Containing Zinc

In the experiment with zinc addition, zinc solution was prepared using ZnO. Zinc oxide was equilibrated in de-ionized water for 1 day. The solution was then filtered through a 0.2- μm pore-size filter to give a stock solution with a concentration of 850 ppb, determined with ICP-OES. The proper amount of zinc stock solution was added to the coolant containing boron and lithium to obtain a zinc concentration of 20 ppb, which was a value in between 5 and 40 ppb Zn used in PWRs.

Appendix C LRS Spectra

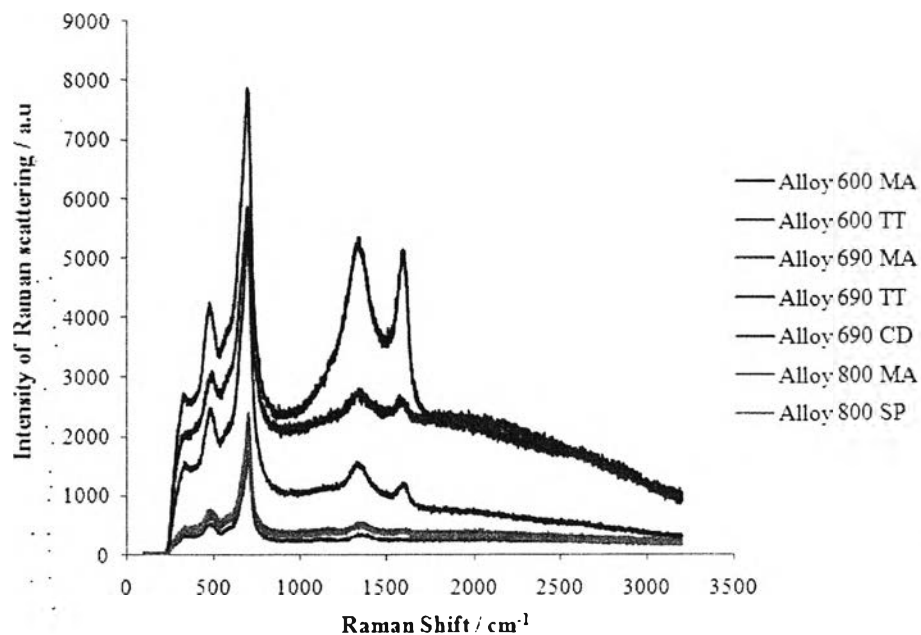


Figure C.1 Raman spectra of crystallites on SG alloy samples in Run 4.

Table C.1 Raman shift of crystallites on SG alloy samples in Run 4

SG alloy	Raman Shift/cm ⁻¹			
Alloy 600 MA	306	496.7	692.2	1371.4
Alloy 600 TT	361.2	481.8	692.2	1347.5
Alloy 690 MA	377.4	499.8	697.3	1384.3
Alloy 690 TT	350.4	484	695.2	1341.95
Alloy 690 CD	331	486.1	699.3	1390.7
Alloy 800 MA	369.8	487.2	699.3	1357.6
Alloy 800 SP	335.3	482.9	700.4	1369.6

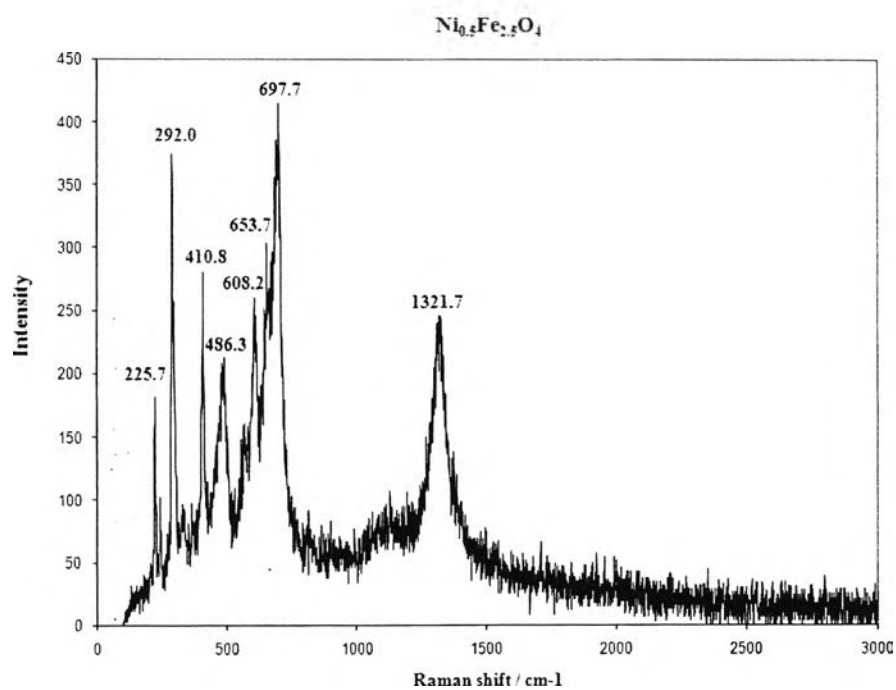


Figure C.2 Raman spectrum of $\text{Ni}_{0.5}\text{Fe}_{2.5}\text{O}_4$.

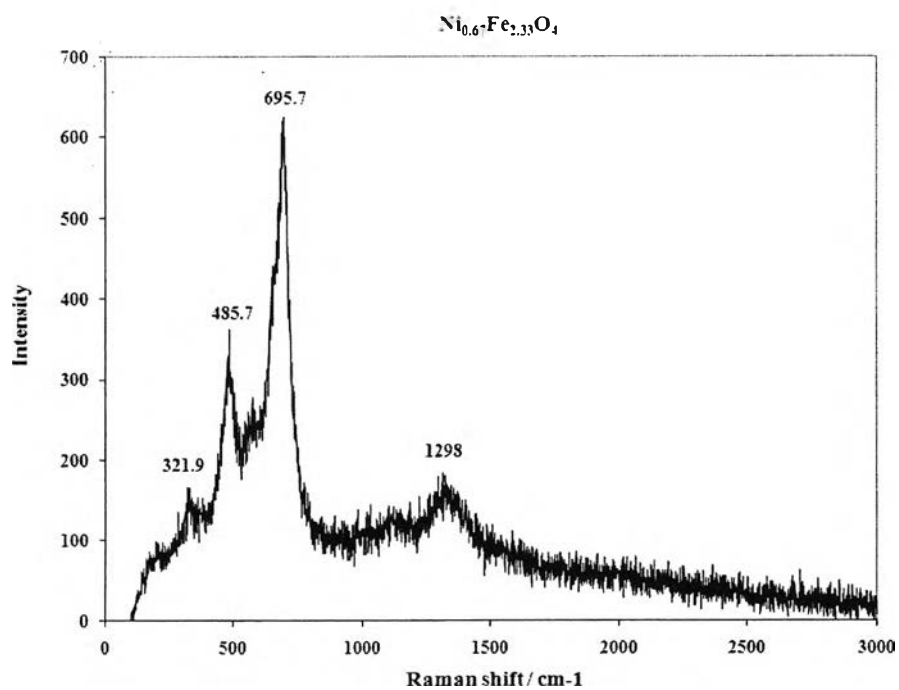


Figure C.3 Raman spectrum of $\text{Ni}_{0.67}\text{Fe}_{2.33}\text{O}_4$.

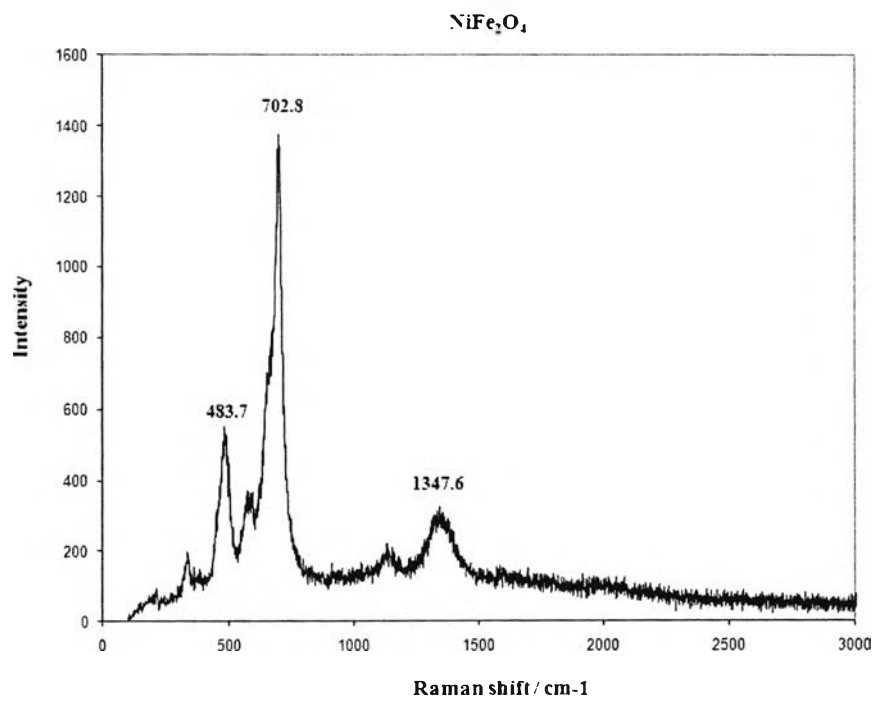


Figure C.4 Raman spectrum of NiFe_2O_4 .

Appendix D Calculation of Corrosion Rate

The corrosion rate of SG alloy sample in this work was calculated from the oxide thickness of the sample. The density of the oxide was assumed to be the density of magnetite (Fe_3O_4), which is 5.2 g/cm^3 . This is an example of the calculation for the corrosion rate of Alloy 600 MA in Run 1.

From the cross-sectional SEM image of SG1 and the XPS depth profile, the oxide thickness of Alloy 600 MA in Run 1 was $\sim 0.1 \mu\text{m}$.

$$\begin{aligned} \text{\%wt of Fe in the oxide} &= \text{Density of Fe}_3\text{O}_4 \times \text{oxide thickness} \times (3 \times \text{M.W. of Fe} / \\ &\quad \text{M.W. of Fe}_3\text{O}_4) \times (1 \text{ cm} / 10^4 \mu\text{m}) \\ &= 5.2 (\text{g/cm}^3) \times 0.1 (\mu\text{m}) \times ((3)(56)/232) \times (1/10^4) (\text{cm}/\mu\text{m}) \\ &= 0.000038 \text{ g/cm}^2 \end{aligned}$$

Since the Alloy 600 MA was exposed to the coolant for 10 days,

$$\begin{aligned} \text{Corrosion rate} &= 0.038 (\text{mg/cm}^2) \times 100 (\text{cm}^2/\text{dm}^2) \times (1/10) (1/\text{day}) \\ &= 0.38 (\text{mg/dm}^2 \cdot \text{day}) \text{ or mdd} \\ &= 1.62 \mu\text{m/yr} \end{aligned}$$

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Proceedings:

1. Luttikul, A., Lister, D.H., Steward, F.R., and Rirksomboon, T. (2010, April 22)
Oxide Film Characteristics under PWR Primary Coolant Conditions. Proceeding
of 1st National Research Symposium on Petroleum, Petrochemicals, and
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