

**Synthesis and Characterization of Polyphase Waste Form:  
 $Gd_3Fe_5O_{12}+(Ni_xMn_{1-x})(Fe_yCr_{1-y})_2O_4$**

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**ABSTRACT**

The synthesis of polyphase waste form, which is an immobilization matrix for the HLW, was performed with the mixed composition of garnet and spinel ( $Gd_3Fe_5O_{12} + (Ni_xMn_{1-x})(Fe_yCr_{1-y})_2O_4$ ) at temperatures in the range of 1200°C to 1400°C. The phases synthesized from all stoichiometric compositions were garnet, perovskite, and spinel. Especially, garnet was synthesized only in the composition of the highest content of Fe ( $y=0.9$ ), whereas it was not synthesized in other compositions. This result indicated that the content of Fe was closely related to the formation of garnet. The composition of garnet revealed that the content of Gd was exceeded and that of Fe was depleted. Preferential distribution of elements in the phases can be attributed to the nonstoichiometric composition of garnet.

**INTRODUCTION**

Glass, such as borosilicates glass, has been studied for a long time as a waste form for radioactive waste. However, glass thermodynamically is unstable and subjected to devitrification. It can result in the decrease of its chemical durability and the escape of radionuclides into the environment. Ringwood et al. (1988) suggested titanate-based ceramics "SYNROC" as an alternative matrix of glass to minimize these disadvantages [1]. Their suggestion based on the fact that some natural minerals containing radioactive elements have retained their structure stable for millions years. Until now, the synthesis and characterization study on various kinds of waste forms has been carried out [1-12].

SYNROC is a multi-phase matrix that includes zirconolite, hollandite, perovskite and titanates. Each phase can accommodate important radionuclides selectively: Hollandite (Cs, Rb, Ba and fission product), zirconolite (U, Zr, Np and Pu), perovskite (Sr and ultrauranic Np and Pu). Tailored ceramics, as an analogue of SYNROC, were developed mainly at the Rockwell International Science Center [13-14]. It was applied to immobilize the defense waste and contained magnetoplumbite, spinel, nepheline, uraninite and corundum.

Major fraction of activity of spent fuel accounts for radionuclides of fission product elements with mass numbers from 85 to 106 and from 125-147 (Kr, Sr, Y, Zr, Tc, Rb, Y, Sb, Cs, Ba, Ce, Pm, etc), unreacted fuel (U), minor actinides (Np, Pu, Am, Cm), and activated products (H, C, Al, Na, Mn, Fe, Co and other) [15]. Such existence of various elements in high level waste requires multi-phase ceramic that can accommodate them selectively.

In this study, we selected the polyphase ceramic, namely,  $Gd_3Fe_5O_{12+(Ni_xMn_{1-x})(Fe_yCr_{1-y})_2O_4}$  ( $x=0.1-0.9$  and  $y=0.1-0.9$ ) that contained garnet considered as a promising waste form and spinel composition, examined phase relation and physicochemical properties of coexisted phases in the compositions and confirmed accommodation relation of elements and phases.

## **EXPERIMENTAL**

The synthesis of polyphase ceramic was performed by the Cold Press and Sintering (CPS) method.  $Gd_2O_3$  (Cerac Co., 3N),  $Fe_2O_3$  (Rare Metallic Co., 3N),  $Cr_2O_3$  (Rare Metallic Co., 3N),  $MnO$  (Rare Metallic Co., 3N) and  $NiO$  (High Purity Chemicals, 3N) were used as starting materials. These powders were mixed in the appropriate ratios,  $Gd_3Fe_5O_{12+(Ni_xMn_{1-x})(Fe_yCr_{1-y})_2O_4}$  ( $x$  and  $y=0.1-0.9$ ) with alcohol, using an alumina mortar and pestle. The mixed powders were pressed into pellets (10-mm diameter x 2-mm height) at a pressure of  $400 \text{ kg/cm}^2$ . The pellets were sintered at  $1200-1400^\circ\text{C}$  for 20 hr. Phase identification was determined by X-ray diffraction (reflection mode,  $\text{CuK}\alpha$  radiation) using a Phillips X'pert MPD X-ray diffractometer attached with a graphite monochromator. The compositions of synthetic phases were analyzed with a SEM/EDS analyzer.

## RESULTS

Before polyphase ceramic was synthesized, its end members, garnet( $\text{Gd}_3\text{Fe}_5\text{O}_{12}$ ) and spinel ( $(\text{Ni}_x\text{Mn}_{1-x})(\text{Fe}_y\text{Cr}_{1-y})_2\text{O}_4$ ) were synthesized, and experimental condition of polyphase was set up based on the results of the synthesis of end members.

### The synthesis of garnet

$\text{Gd}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ , and perovskite were observed at  $1000^\circ\text{C}$ . At  $1100^\circ\text{C}$ , garnet formed instead of disappearing  $\text{Gd}_2\text{O}_3$  and the diffraction intensities of  $\text{Fe}_2\text{O}_3$  and perovskite distinctly decreased.  $\text{Fe}_2\text{O}_3$  and perovskite disappeared with increase of temperature. The single phase of garnet was synthesized at  $1300^\circ\text{C}$  and the XRD pattern of specimen did not change up  $1400^\circ\text{C}$  (Fig. 1a). Finally the specimen was melted at  $1500^\circ\text{C}$ . Accordingly, it was confirmed that the optimal synthetic temperature of garnet was  $1300^\circ\text{C}$ . Through phase identification, formation reaction of garnet can be shown below:



In the results of SEM/EDS analysis, garnet observed in matrix, coexisted with minor  $\text{Fe}_2\text{O}_3$ . The composition of garnet was  $\text{Gd}_{3.1}\text{Fe}_{4.9}\text{O}_{12}$  similar to stoichiometric one, although the content of Gd was slightly excessive, whereas that of Fe was depleted.

### The synthesis of spinel

We selected  $(\text{Ni}_x\text{Mn}_{1-x})(\text{Fe}_y\text{Cr}_{1-y})_2\text{O}_4$  as the composition of spinel considering the composition of liquid HLW from reprocessing and carried out the synthetic experiment of spinel (Fig. 1b). In the results of XRD analysis, single phases of spinels were observed in the temperature range of  $1100$  and  $1300^\circ\text{C}$  except the composition of  $x=0.1$  and  $y=0.9$ . In low temperatures, coexisted phases were closely related to the stoichiometric compositions, especially the mixed oxides of  $\text{Cr}_2\text{O}_3$  and  $\text{Fe}_2\text{O}_3$  were always observed at  $y=0.5$ . It was confirmed that the compositions of spinels were similar to stoichiometric compositions.

### The synthesis of multiphase ceramics

As described above, single phase of garnet and spinel formed at  $1300^\circ\text{C}$  and  $1100$ - $1300^\circ\text{C}$ , respectively. From these results, we performed the synthetic experiment of polyphase ceramic in

the temperature range of 1200-1400°C. Three types of phase series observed: Fe<sub>2</sub>O<sub>3</sub>-perovskite-spinel, Fe<sub>2</sub>O<sub>3</sub>-spinel-garnet and perovskite-spinel-garnet (Table I). Garnet was formed only in the composition with the highest content of Fe (y=0.9) (Table I and Fig. 2) and the diffraction intensity of spinel was directly proportional to the content of Ni (Fig. 3). Accordingly, these results indicated that the content of Fe and Ni played an important role in the formations of garnet and spinel.

Fig. 4 shows the back scattered electron (BSE) images of specimens sintered at 1200-1400°C. In all specimens, garnet, perovskite or spinel observed as an aggregate or island form. The analytical data for phases in specimens is shown in Table II. Spinel or Fe<sub>2</sub>O<sub>3</sub> which was identified through XRD analysis was not found except the compositions of x=0.5 and y=0.1, and x=0.9 and y=0.5. It resulted from the similarity of brightness of such phases, and especially, phases with relative low diffraction intensity were not observed. Not being presented in this paper, but phases not detected in the XRD data were observed in the element distribution map through the EDS analysis.

The composition of garnet was nonstoichiometric as shown in Table II. And the composition of spinel which was coexisted with garnet was similar to the stoichiometric composition, whereas that of spinel not coexisted with garnet was irregular. In contrast with the composition of garnet and spinel, perovskite had relative regular composition as Gd-Fe-Cr oxides in all range of composition (Table II).

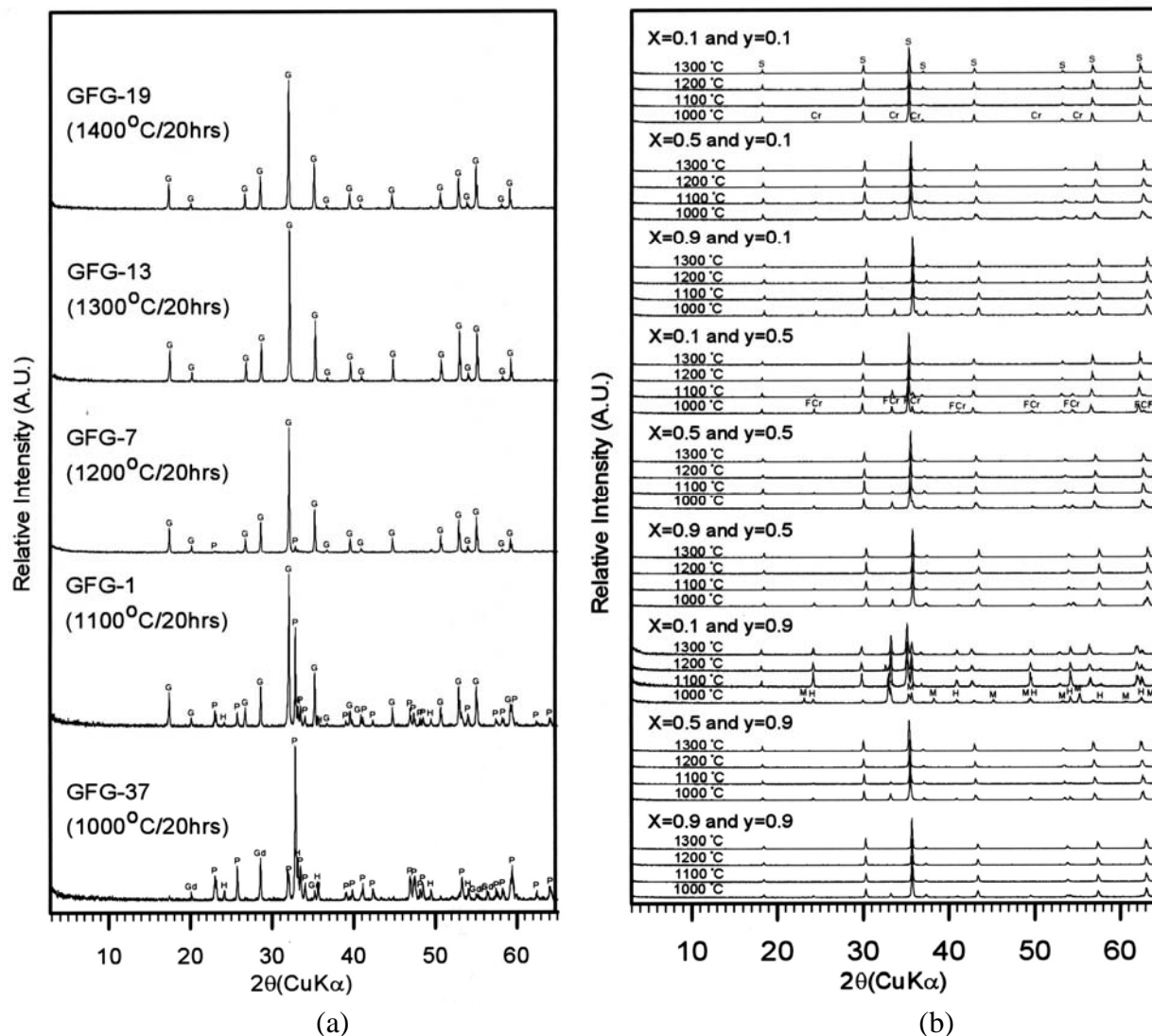


Fig. 1. XRD patterns of (a) garnet (Gd<sub>3</sub>Fe<sub>5</sub>O<sub>12</sub>) and (b) (Ni<sub>x</sub>Mn<sub>1-x</sub>)(Fe<sub>y</sub>Cr<sub>1-y</sub>)<sub>2</sub>O<sub>4</sub> (x=0.1-0.9, y=0.1-0.9). G: garnet, P: perovskite, H: Fe<sub>2</sub>O<sub>3</sub>, M: Mn<sub>2</sub>O<sub>3</sub>, Gd: Gd<sub>2</sub>O<sub>3</sub>, Sp: spinel, FCr: (Fe<sub>0.6</sub>Cr<sub>0.4</sub>)<sub>2</sub>O<sub>3</sub>, Cr: Cr<sub>2</sub>O<sub>3</sub>.

Table I. Relative Intensities of Phases synthesized with the Batch Composition,  $Gd_3Fe_5O_{12}+(Ni_xMn_{1-x})(Fe_yCr_{1-y})_2O_4$ .

No.	Conditions			H	P	Sp	G
	Temp.	Hr	Atm.	2.695	2.72	2.53	2.79
$Gd_3Fe_5O_{12}+(Ni_{0.1}Mn_{0.9})(Fe_{0.1}Cr_{0.9})_2O_4$							
GDS-35	1200	20	O2	25	100	10	-
GDS-31	1300	20	O2	23	100	8	-
GDS-39	1400	20	O2	18	100	9	-
$Gd_3Fe_5O_{12}+(Ni_{0.5}Mn_{0.5})(Fe_{0.1}Cr_{0.9})_2O_4$							
GDS-3	1200	20	O2	17	100	27	-
GDS-8	1300	20	O2	17	100	23	-
GDS-13	1400	20	O2	15	100	21	-
$Gd_3Fe_5O_{12}+(Ni_{0.9}Mn_{0.1})(Fe_{0.1}Cr_{0.9})_2O_4$							
GDS-37	1200	20	O2	11	100	38	-
GDS-33	1300	20	O2	12	100	33	-
GDS-41	1400	20	O2	11	100	32	-
$Gd_3Fe_5O_{12}+(Ni_{0.1}Mn_{0.9})(Fe_{0.5}Cr_{0.5})_2O_4$							
GDS-5	1200	20	O2	21	100	8	-
GDS-10	1300	20	O2	21	100	7	-
GDS-15	1400	20	O2	27	100	10	-
$Gd_3Fe_5O_{12}+(Ni_{0.5}Mn_{0.5})(Fe_{0.5}Cr_{0.5})_2O_4$							
GDS-2	1200	20	O2	16	100	17	-
GDS-7	1300	20	O2	29	100	24	-
GDS-12	1400	20	O2	19	100	18	-
$Gd_3Fe_5O_{12}+(Ni_{0.9}Mn_{0.1})(Fe_{0.5}Cr_{0.5})_2O_4$							
GDS-4	1200	20	O2	13	100	34	-
GDS-9	1300	20	O2	13	100	35	-
GDS-14	1400	20	O2	20	100	41	-
$Gd_3Fe_5O_{12}+(Ni_{0.1}Mn_{0.9})(Fe_{0.9}Cr_{0.1})_2O_4$							
GDS-36	1200	20	O2	4		39	100
GDS-32	1300	20	O2	8		45	100
GDS-40	1400	20	O2	-	12	41	100
$Gd_3Fe_5O_{12}+(Ni_{0.5}Mn_{0.5})(Fe_{0.9}Cr_{0.1})_2O_4$							
GDS-1	1200	20	O2	-	3	16	100
GDS-6	1300	20	O2	-	7	17	100
GDS-11	1400	20	O2	-	40	28	100
$Gd_3Fe_5O_{12}+(Ni_{0.9}Mn_{0.1})(Fe_{0.9}Cr_{0.1})_2O_4$							
GDS-38	1200	20	O2	18	100	29	-
GDS-34	1300	20	O2	24	100	39	-
GDS-42	1400	20	O2	19	100	31	-

Abbreviations: G: garnet, P: perovskite, H:  $Fe_2O_3$ , Gd:  $Gd_2O_3$ , Sp: spinel

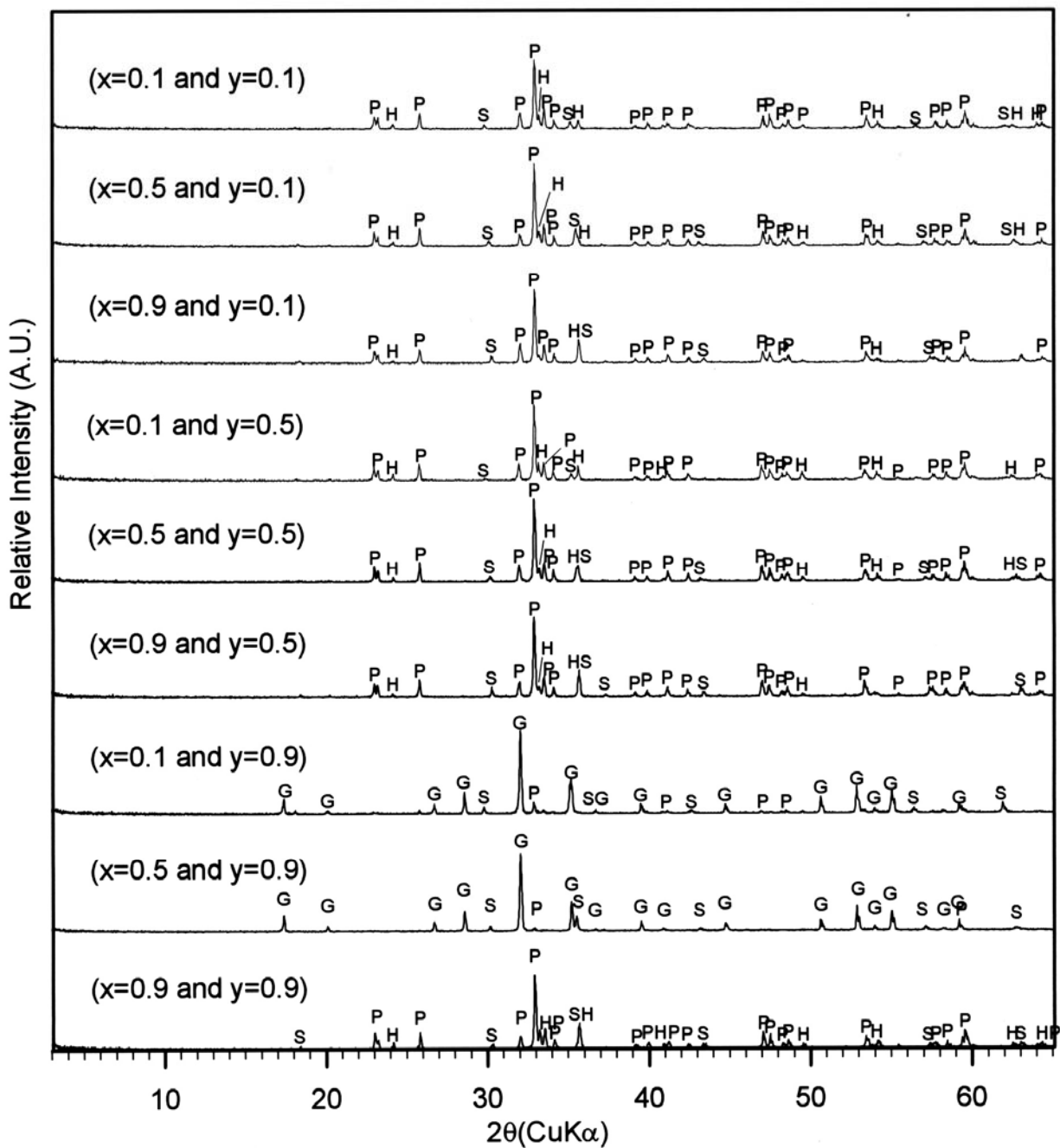


Fig. 2. XRD patterns of  $Gd_3Fe_5O_{12} + (Ni_xMn_{1-x})(Fe_yCr_{1-y})_2O_4$  sintered at optimal conditions. G: garnet, P: perovskite, H:  $Fe_2O_3$  and S: spinel.

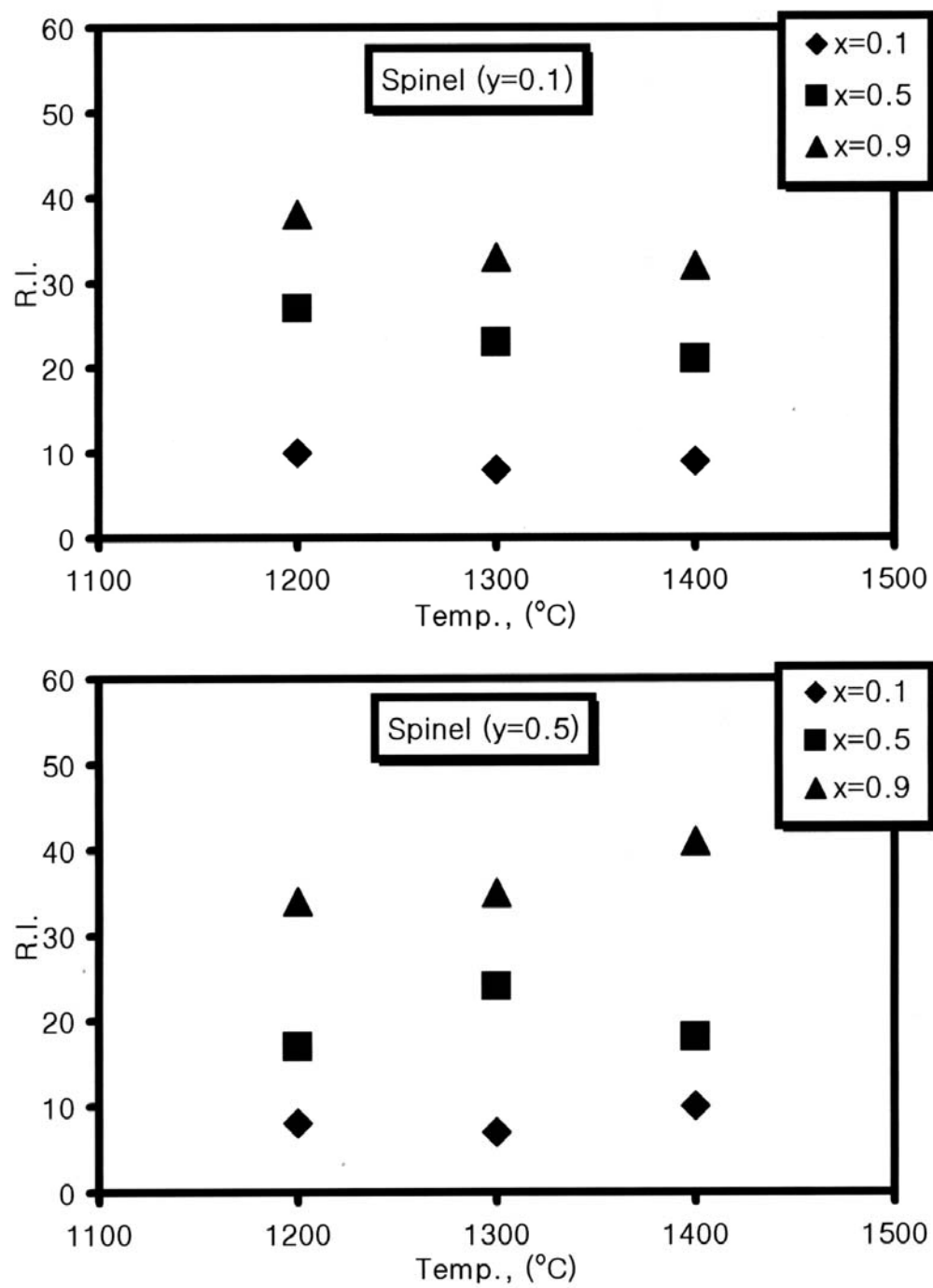


Fig. 3. The relation of relative diffraction intensity with the content of Ni (x) and Fe (y).



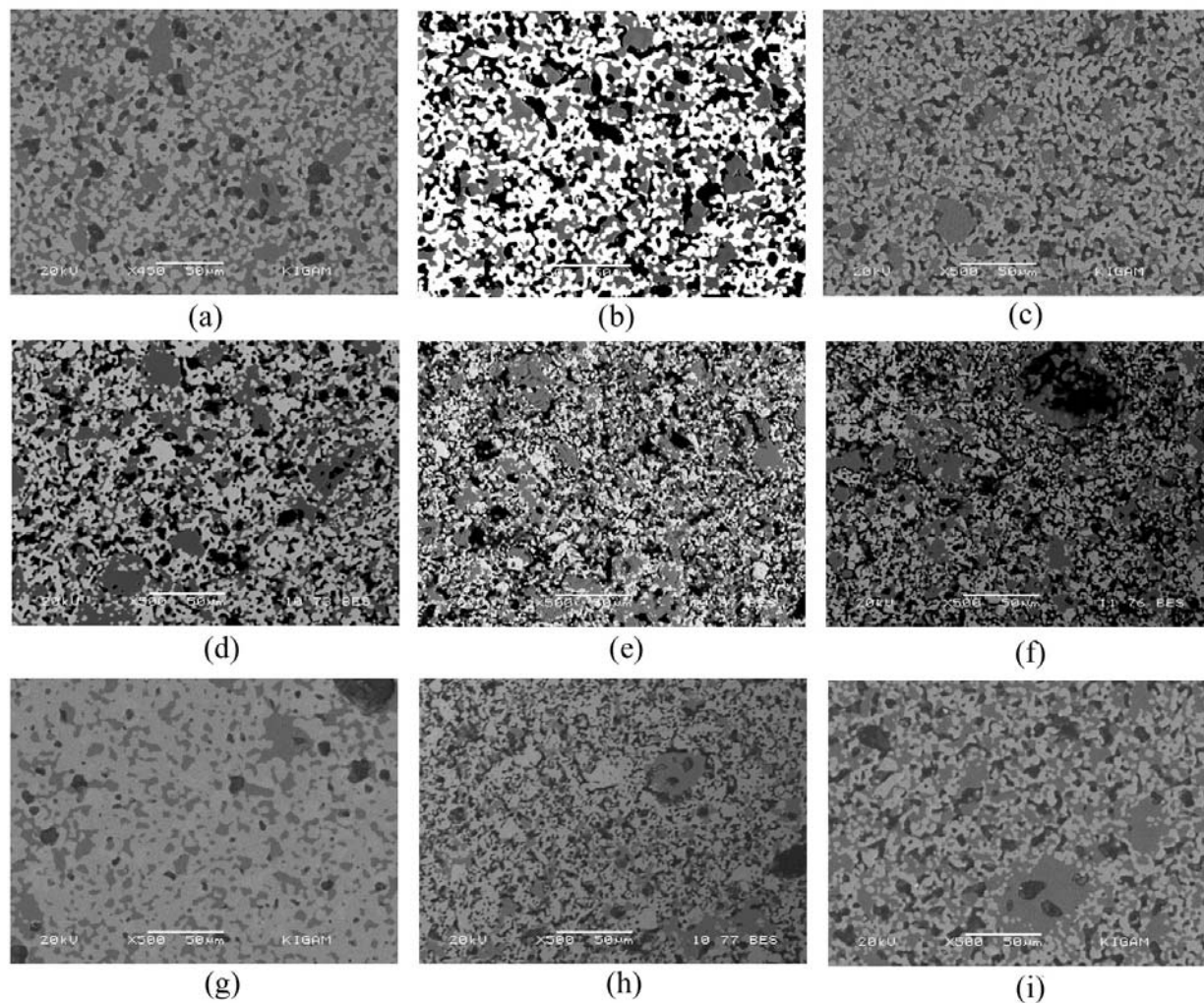


Fig. 4. Back scattered electron (BSE) images of specimens synthesized from the composition of  $Gd_5Fe_3O_{12} + (Ni_xMn_{1-x})(Fe_yCr_{1-y})_2O_4$ . (a) GDS-39 ( $x=0.1, y=0.1, 1400^\circ C, 20hr$ ), (b) GDS-13 ( $x=0.5, y=0.1, 1400^\circ C, 20hr$ ), (c) GDS-41 ( $x=0.9, y=0.1, 1400^\circ C, 20hr$ ), (d) GDS-10 ( $x=0.1, y=0.5, 1400^\circ C, 20hr$ ), (e) GDS-2 ( $x=0.5, y=0.5, 1200^\circ C, 20hr$ ), (f) GDS-4 ( $x=0.9, y=0.5, 1200^\circ C, 20hr$ ), (g) GDS-40 ( $x=0.1, y=0.9, 1400^\circ C, 20hr$ ), (h) GDS-1 ( $x=0.5, y=0.9, 1200^\circ C, 20hr$ ), (i) GDS-42 ( $x=0.9, y=0.9, 1400^\circ C, 20hr$ ). White (Perovskite or garnet); Gray (Spinel or  $Fe_2O_3$ ); Black (Holes)

Table II. Chemical Formula of Phases which were contained Matrices synthesized with the Precursor Composition of  $Gd_3Fe_5O_{12}+(Ni_xMn_{1-x})(Fe_yCr_{1-y})_2O_4$ .

x	y	Garnet	Spinel	Perovskite	Fe <sub>2</sub> O <sub>3</sub>
0.1	0.1	-	-	Gd <sub>0.9</sub> (Fe <sub>0.7</sub> Cr <sub>0.4</sub> )O <sub>3</sub>	(Fe <sub>1.5</sub> Mn <sub>0.1-0.2</sub> Cr <sub>0.4</sub> )O <sub>3</sub>
0.5	0.1	-	(Ni <sub>0.5-0.6</sub> Mn <sub>0.4-0.5</sub> )(Fe <sub>0.8</sub> Cr <sub>0.2</sub> ) <sub>2</sub> O <sub>4</sub>	Gd <sub>1.0</sub> (Fe <sub>0.7</sub> Cr <sub>0.3</sub> )O <sub>3</sub>	(Fe <sub>1.6</sub> Mn <sub>0.1</sub> Cr <sub>0.3</sub> )O <sub>3</sub>
0.9	0.1	-	(Ni <sub>0.5</sub> Mn <sub>0.1</sub> )(Fe <sub>0.9</sub> Cr <sub>0.2-0.3</sub> ) <sub>2</sub> O <sub>4</sub>	Gd <sub>0.9</sub> (Fe <sub>0.7-0.8</sub> Cr <sub>0.3-0.4</sub> )O <sub>3</sub>	-
0.1	0.5	-	-	Gd <sub>1.0</sub> (Fe <sub>0.8</sub> Cr <sub>0.2</sub> )O <sub>3</sub>	(Fe <sub>1.7</sub> Mn <sub>0.1</sub> Cr <sub>0.2</sub> )O <sub>3</sub>
0.5	0.5	-	-	Gd <sub>1.0</sub> (Fe <sub>0.7-0.8</sub> Cr <sub>0.2</sub> )O <sub>3</sub>	(Fe <sub>1.7-1.8</sub> Mn <sub>0.1</sub> Cr <sub>0.1-0.2</sub> )O <sub>3</sub>
0.9	0.5	-	(Ni <sub>0.6</sub> Mn <sub>0.1</sub> )(Fe <sub>1.0</sub> Cr <sub>0.1</sub> ) <sub>2</sub> O <sub>4</sub>	Gd <sub>1.0</sub> (Fe <sub>0.8-0.9</sub> Cr <sub>0.1-0.2</sub> )O <sub>3</sub>	-
0.1	0.9	Gd <sub>2.7-2.8</sub> Fe <sub>5.1</sub> O <sub>12</sub> (Cr: 0.2)	(Ni <sub>0.1</sub> Mn <sub>0.7</sub> )(Fe <sub>1.1</sub> Cr <sub>0.05</sub> ) <sub>2</sub> O <sub>4</sub>	-	-
0.5	0.9	Gd <sub>3.4</sub> Fe <sub>4.4</sub> O <sub>12</sub> (Cr: 0.2)	(Ni <sub>0.5</sub> Mn <sub>0.4-0.5</sub> )(Fe <sub>1.0</sub> Cr <sub>0.05</sub> ) <sub>2</sub> O <sub>4</sub>	Gd <sub>1.0</sub> (Fe <sub>0.9</sub> Cr <sub>0.1</sub> )O <sub>3</sub>	-
0.9	0.9	-	(Ni <sub>0.6-0.7</sub> Mn <sub>0.1</sub> )(Fe <sub>0.9</sub> Cr <sub>0.2</sub> ) <sub>2</sub> O <sub>4</sub>	Gd <sub>0.9</sub> (Fe <sub>0.7</sub> Cr <sub>0.4</sub> )O <sub>3</sub>	-

## DISCUSSION

Although the composition which was able to coexist with garnet and spinel was selected, they were only formed at the compositions ( $x=0.1-0.5$  and  $y=0.9$ ). Except such compositions perovskite had the strongest intensity in most of compositions, and the intensity of perovskite dramatically decreased in the formation of garnet. It was similar to the result of synthesis of pure garnet ( $Gd_3Fe_5O_{12}$ ).

Garnet and spinel had nonstoichiometric compositions and it can be interpreted as a preferential distribution of elements in each phase. Namely, Fe was not only the major elements of garnet and perovskite but also those of spinel. Also Gd was the major elements of garnet and perovskite, but it was not related with the formation of spinel. In the result, the nonstoichiometric composition of garnet was due to coexistence with Gd-Fe perovskite not considered in the stoichiometric composition. Also nonstoichiometric composition of spinel which was not coexisted with garnet was related to the formation of perovskite. Namely, the reaction equation of garnet to perovskite was:  $Gd_3Fe_5O_{12} = 3GdFeO_3 + Fe_2O_3$ . If all Gd of garnet were distributed to the perovskite, 1 mole Fe<sub>2</sub>O<sub>3</sub> has to be done in a phase. In the result, this content of Fe<sub>2</sub>O<sub>3</sub> was distributed to spinel, and the content of Fe in spinel increases. The existence of Cr in perovskite is based on the fact that Fe (0.645Å) and Cr (0.615Å) which were major elements of perovskite

had same valence (+3) and their ionic radii was more similar than other elements ( $\text{Ni}^{+2}$ : 0.69Å and  $\text{Mn}^{+2}$ : 0.83Å).

## CONCLUSION

Certain types of defense HLW streams at Hanford contained significant concentrations of metal ions such as Cr, Ni and Fe. In this study, polyphase waste form which was the mixture of  $\text{Gd}_3\text{Fe}_5\text{O}_{12}$  and  $(\text{Ni}_x\text{Mn}_{1-x})(\text{Fe}_y\text{Cr}_{y-1})_2\text{O}_4$  was synthesized using Gd as a imitator of Pu. Also, we are intended to examine phase relation and physicochemical properties of coexisted phases in the compositions and to confirm accommodation relation of elements and phases.

The synthetic experiment of polyphase ceramic was performed in the temperature range of 1200-1400°C. Three types of phase series observed:  $\text{Fe}_2\text{O}_3$ -perovskite-spinel,  $\text{Fe}_2\text{O}_3$ -spinel-garnet and perovskite-spinel-garnet. Garnet was formed only in the composition with the highest content of Fe ( $x=0.1-0.5$  and  $y=0.9$ ). The formation of Gd-Fe perovskite and preferential distribution of elements in each phase can be attributed to the nonstoichiometric composition of garnet and spinel.

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