SYNTHESIS OF A NEW Y-Fe-O ULTRAFINE PARTICLES USING INDUCTIVELY COUPLED PLASMA

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ABSTRACT

A new phase in Y-Fe-O system was synthesized by RF thermal plasma evaporation method using a co-precipitated powder or Y and Fe mixed nitrate solutions. This presentation reports synthesizing the unknown of a pure single phase and characterizing it.

INTRODUCTION

Ultrafine particles of multi-component oxide can be synthesized by introducing powders or aqueous solutions of metal salts into RF thermal plasma[1][2]. But multi-phase products often appear owing to differences in vapor pressures of components, and the experimental parameters must be controlled to obtain a multi-component oxide of single phase that has the objective stoichiometry. In Y-Fe-O system, several compounds are known such as orthorhombic $YFeO_3$(o-FeO_3),garnet($Y_3Fe_5O_{12}$:YIG) and metastable hexagonal $YFeO_3$(h-$YFeO_3$)[3][4]. In a series of experiments synthesizing the compounds in Y-Fe-O system by RF thermal plasma evaporation method, a new phase in Y-Fe-O system was obtained as the main product which had a XRD pattern not found in JCPDS, and the products containing the unknown phase were attracted to a magnet. This presentation reports synthesizing the unknown as a pure single phase and characterizing it.

EXPERIMENTAL

The experimental apparatus is shown in Fig. 1. The plasma was generated with a three-turn work coil coupled to a RF power supply(4MHz in frequency and 60kW in maximum plate power).

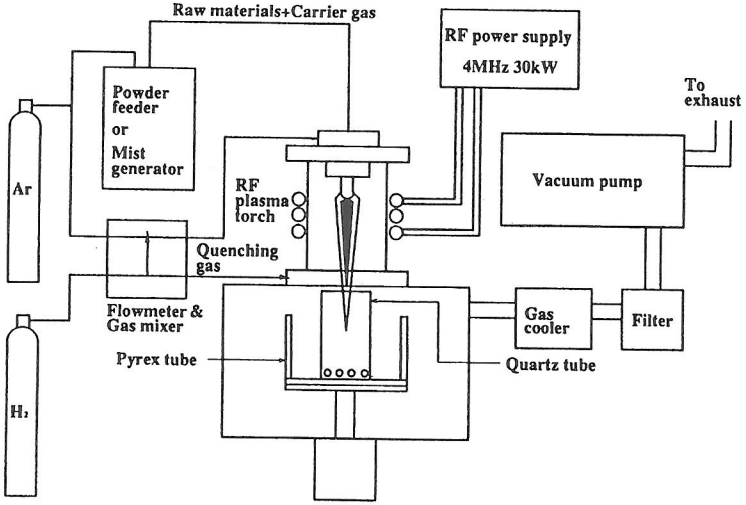
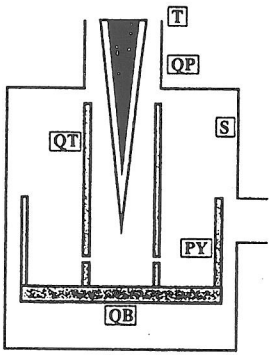


Fig. 1 Experimental apparatus

A microfeeder was used to supply powder raw material. Liquid raw materials were supplied by a mist generator equipped with a ultrasonic vibrator of 2.5MHz. A flask containing a liquid was placed just above the vibrator. These raw materials were supplied with Ar carrier gas from the top of plasma along plasma center axis. A copper nozzle was placed to introduce the powder into plasma.Liquids were introduced through thin quartz tubes into plasma. The plasma tail flame was enclosed by a quartz tube and a pyrex tube to keep the temperature of tail flame high. Two quartz tubes having different inner diameters(ID) were used to control the heat radiation of plasma tail flame. Experiments were done with Ar-O_2 plasma. The powder raw material was prepared by co-precipitation using 1N NH_4OH and mixed nitrate solutions of Y:Fe(molar)=3:5. The liquid raw materials were mixed Y and Fe nitrate solutions. Y:Fe ratios(molar) were 1:2,1:2.5,1:3 and 1:4.



T : Torch wall QP : Quenching port
QT : Quartz tube QB : Quartz board
PY : Pyrex tube S : Side

Fig. 2 Product collection positions

The total metal concentration was 0.4M.

The products were collected from the positions shown in Fig. 2. The collection area of QT was divided into QT-upper,QT-middle and QT-lower. These are shown as QTU,QTM and QTL.

The experimental conditions are summarized in Table 1 for the cases of using powder and liquid raw materials. The products were characterized by TEM and XRD.

Table 1 Experimental conditions

	Powder	Liquid
Plasma power (kW) (plate)	32.4	20.8
Plasma gas Ar (l/min)	91~93	49
O_2 (l/min)	20	26
Powder feed rate	1.0 g/min	0.17~0.28 ml/min
Powder feed time (min)	24	40~60

RESULTS AND DISCUSSION

Fig. 3 shows the XRD patterns of products collected from QTU,QTM and QTL in the experiment using the powder raw material. Y_2O_3,h-$YFeO_3$ and an unknown phase were found. The unknown phase had a XRD pattern not found in JCPDS. The XRD pattern showed that its crystal structure was probably face-centered cubic with a_0=0.485nm. The particle size distribution was measured from TEM observation of the product collected from QTM. The mean particle diameter was 20nm, of which standard deviation was found to be 1.5.

As the products containing the unknown phase were attracted to a magnet, the phase was presumed to be a ferromagnetic(or ferrimagnetic) compound, and it was stable up to 1273K in atmospheric heat treatment.

Fig. 4 shows XRD pattrns of products collected from QTU in the experiments using liquid raw materials having various Y:Fe ratios.

In the case of Y:Fe=1:2 and 1:2.5 the existence of $Y_3Fe_5O_{12}$ and the unknown phase was confirmed obviously. But the products synthesized from liquids having Y:Fe=1:3 and 1:4 did not contain $Y_3Fe_5O_{12}$. The unknown of almost pure single phase was synthesized from the liquid having Y:Fe=1:3. In another experiment using Ar plasma, the product collected from QTU contained only γ-Fe_2O_3 and h-$YFeO_3$. These results showed that the optimum Y:Fe ratio exists to synthesize the unknown powder as a pure single phase.

TEM observation of the product synthesized from the liquid having Y:Fe=1:3 showed that the primary particles of the unknown powder had the diameters of about 10nm.

Fig. 5 shows the XRD patterns of products collected from QTU in the experiments using two quartz tubes having different inner diameters(A:ID=93mm,B:ID=134mm). The unknown's XRD peaks in case B were broader than those in case A. As the plasma tail flame was close to the inner wall of the quartz tube in case A, much radiation heat was transferred from the tail flame to the deposited product compared with case B. It was presumed that this much radiation heat made the primary particle size large and/or the product's crystallinity good, and the half widths of XRD peaks became small due to these factors in case A. From this result, controlling the radiation heat from the tail flame to the deposited product was shown necessary to obtain the unknown powder as a pure single phase.

Fig. 6 shows XRD patterns of the as-deposited unknown powder synthesized from the liquid raw material having Y:Fe=1:3 and the unknown powder after heated at 1173K for 8hr. The as-deposited powder was not attracted to a magnet. This result was different from those of the products synthesized from the powder raw material. But the powder after heat treatment was attracted to a magnet. The half widths of XRD peaks became small after heat treatment, and this magnetic property was probably due to primary particle size and/or good crystallinity. As the peaks of α-Fe_2O_3 appeared in the powder after heat treatment, a little amorphous Fe_2O_3 was presumed to exist in the as-deposited powder originally.

The detail magnetic characteristics will be shown on ISPC-12.

CONCLUSION

A new phase in Y-Fe-O system was synthesized by RF thermal plasma evaporation method.

From the results of the experiments, controlling Y:Fe ratio of raw material and the radiation heat of plasma tail flame were shown necessary to synthesize the unknown powder as a pure single phase.

Heating the unknown powder of almost pure single phase at 1173K for 8hr made it clear that the unknown powder was attracted to a magnet due to primary particle size and/or good crystallinity.

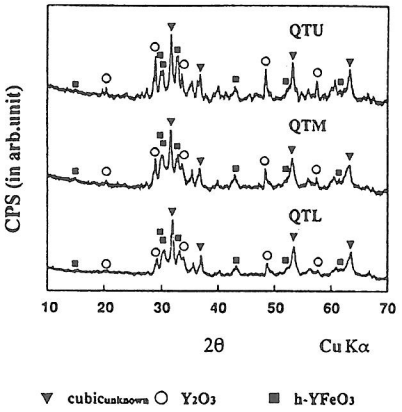


Fig. 3 XRD patterns of products synthesized from the powder (ID=93mm)

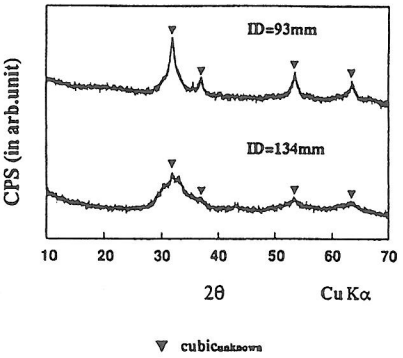


Fig. 5 XRD patterns of products in the experiments using two quartz tubes having different inner diameters

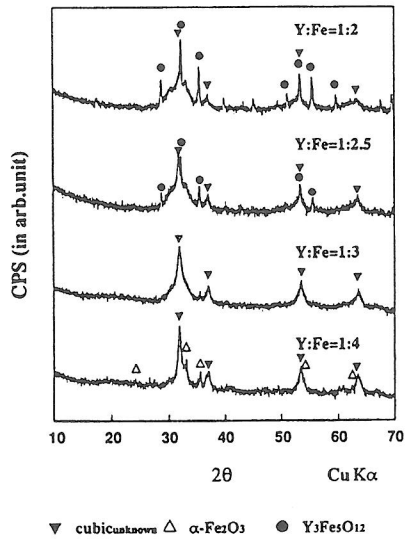


Fig. 4 XRD patterns of products synthesized from the liquids having various Y:Fe ratios (ID=93mm)

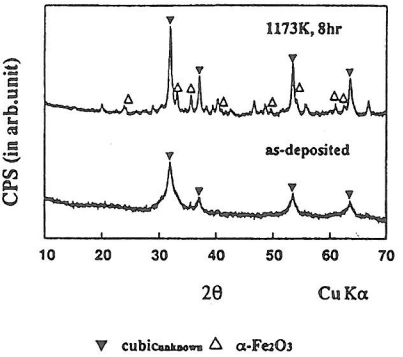


Fig. 6 XRD patterns of the as-deposited unknown powder and the unknown powder after heated at 1173K for 8hr

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