CHAPTER III

SOME EXPERIMENTAL RESULTS

The samples used in this study were prepared in the HIGH TECHNOLOGY CERAMIC LABORATORY of the Department of Physics, Faculty of Science, Mahidol University. Measurements of some properties of the resulting YIG specimens were carried out at Chulalongkorn University and at the above laboratory.

3.1 Fabrication of Yttrium Iron Garnets.

Ultra highgrade oxides were used to fabricate $Y_3Fe_5O_{12}$ 99.99 % purity Y_2O_3 was obtained from Alrich Chemical Company (USA) by Department of Physics, Faculty of Science, Chulalongkorn University and 99.99 % purity Fe_2O_3 was obtained from Johnson & Mattley (UK) by the Department of Physics, Faculty of Science, Mahidol University. The amounts of chemical needed to yield 10 grams of YIG were:

compound	percent purity	molecular weight(g)	weight (g)	characteristic
Y_2O_3	99.99	225.807	6.204	is a fine powder and white
Fe_2O_3	99.99	159.691	7.313	is a fine powder and red

Each time, 6.204 gms of Y_2O_3 and 7.313 gms of Fe_2O_3 were mixed together in an aggate motar. The mixtures were then hand ground into a fine powder. After this grinding, the powder were placed into a platinum crucible, which inturn was placed into a Lindberg Crucible Type Furnace (USA). (see Figure 3.1)

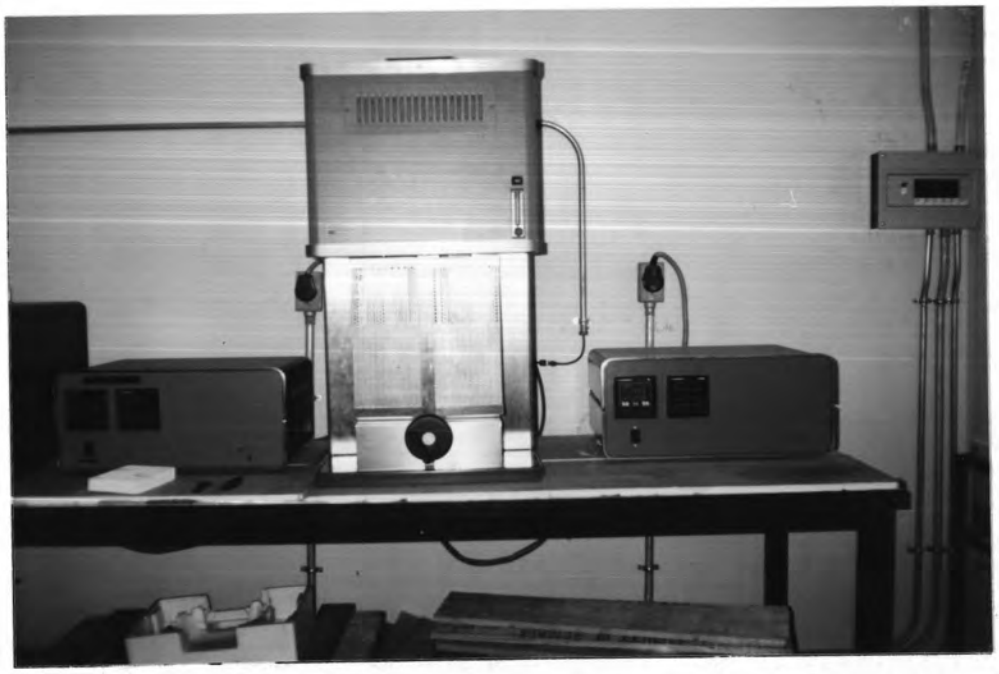


Figure 3.1 Lindberg Crucible Type Furnace.

Several different prefiring temperature were used. The temperature finally adopted was 1380°C. The setting (for the runs reported in this work) on controller for the Lindberg Furnace are:

Ramp	3.3° C/min increasing
Level	1380° C
Dwell	2 hours
Ramp	2° C/min decreasing
Level	110° C
Dwell	1 hour
Oxygen Flow	4 CFH

After firing, the mixture was gray and hard. (Several different dwell times and temperature levels were used in other runs.) Polyvinyl alcohol (0.1377 gms) was added to the calcined mixture and were reground by hand in the aggate motar to a fine powder. The alcohol was added to serve as a binder. The use of the aggate motar is emphasized to point out that the introduction of additional iron (from the use of steel milling jars) during the regrounding process in the standard production of YIG is not a problem here. The mixed powder were then pressed into pellets under a pressure of 2200 psi or 5 tons of force. (see Figure 3.2) These pellets were then put into the Lindberg Furnace for sintering. The settings on the controller for the production of the YIG whose measurements are reported in this thesis are:

Ramp	3.3° C/min increasing
Level	330° C
Dwell	3 hours
Ramp	3.3° C/min increasing
Level	1500° C

Dwell	24 hours
Oxygen Flow	4 CFH

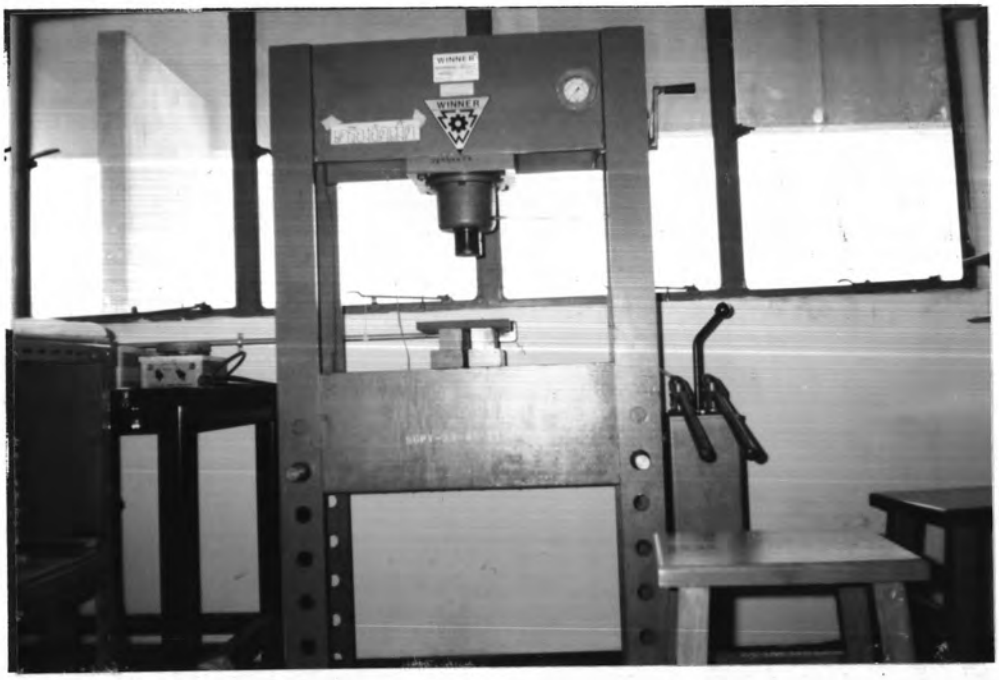


Figure 3.2 Press.

The first ramp step was introduced to drive off the polyvinyl alcohol before the powder underwent its densification during the sintering step. The resulting pellets were gray but were gray-green when they were reground for X-ray and Mössbauer study. In the picture below, we see the grains in a YIG pellet annealed at 1450°C for twenty four hours.

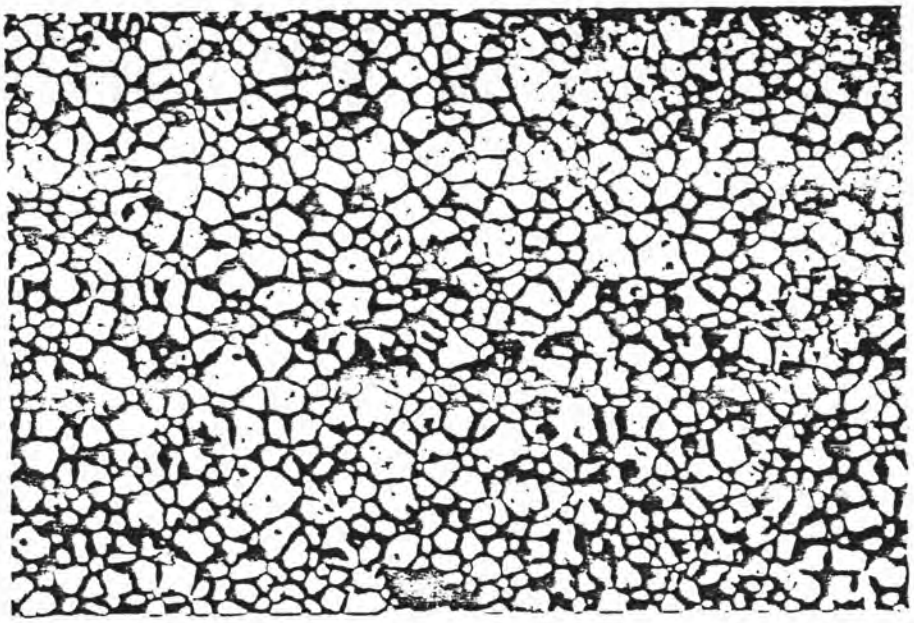


Figure 3.3 Microstructure of yttrium iron garnet sintered at 1450° C for twenty hours. Magnification 1000X using a Nikon Metallurgical Microscope (Mahidol University).

3.2 X-ray Powder Diffraction Study.

To have meaningful measurements, the specimens whose properties are to be measured should be characterized as much as possible. Since the densification of ceramic materials (of which YIG is one) depends on the fabrication processes, the lattice parameters of the YIG are not the same for all YIG. The first measurements we have done is to characterize the crystal structure. We have used the X-ray diffractometers at the Department of Geology, Faculty of Science, Chulalongkorn University (see Figure 3.4) and at the Scientific and Technological Research Equipment Centre, Chulalongkorn University.

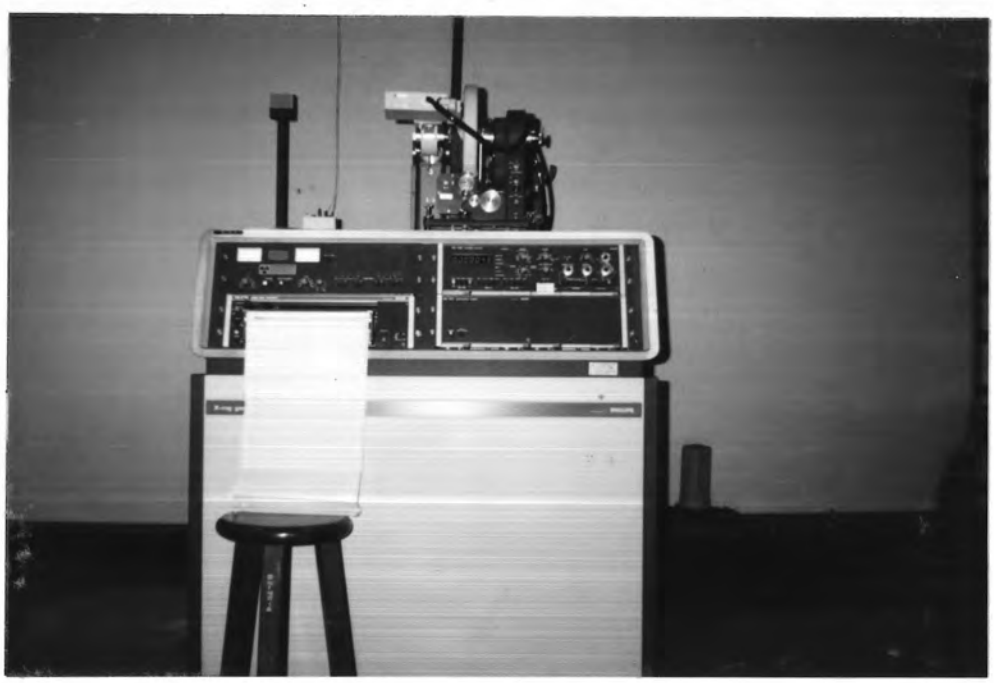


Figure 3.4 X-ray diffractometer of the Geology Department.

Table 3.1

Data File for Yttrium Iron Garnet.

18-1472

d	2.77	3.10	1.65	5.05	$Y_3Fe_2(FeO_4)_3$
I/I_1	100	70	65	6	Yttrium Iron Oxide

Rad. $CuK\alpha_1$ λ 1.5405 Filter Ni Dia.
Cut off I/I_1 Diffractometer
Ref. B. Greenberg, Polytechnic Inst. of Brooklyn, Brooklyn, New York, (1966)

Sys. Cubic S.G. Ia3d (230)
a_0 12.376 b_0 c_0 A C
α β γ Z D_x
Ref. J. Phys. Chem. Solids 3 30 (1957)

εα nωβ εγ Sign
2V D mp Color
Ref.

d A	I/I_1	hkl	d A	I/I_1	hkl
5.05	6	211	1.304	2	851,754
3.30	4	321	1.2139	2	1020,862
3.095	70	400	1.1804	2	1031,952
2.768	100	420	1.1491	16	1040,864
2.526	40	422	1.1298	10	1042
2.427	6	431	1.1024	2	1121,1051
2.261	8	521	1.0942	6	880
2.189	2	440	1.0313	6	1200,884
2.009	10	611,532	1.0175	2	1220
1.786	30	444	1.0039	8	1222,1064
1.716	35	640	0.9606	2	1163,992+
1.683	4	721,633+	.9332	4	1244
1.654	65	642	.9226	16	1260,1084
1.573	2	732,651	.9124	6	1262
1.547	16	800			
1.500	2	820			
1.364	16	840			
1.350	20	842			
1.334	2	921,761			
1.319	16	664			

The data file for the yttrium iron garnet is presented in Table 3.1. The X-ray diffraction pattern of one of the fabricated specimens taken on the X-ray diffractometer in the Scientific and Technological Research Equipment Centre of Chulalongkorn University is shown in Figure 3.5. Most of the X-ray studies were done on the X-ray diffractometer in the Department of Geology, Faculty of Science, Chulalongkorn University. Unlike the Scientific and Technological Research Equipment Centre, use of the instrument in the Geology Department requires that the sample preparation be done by student. Thus, I proceeded as follows:

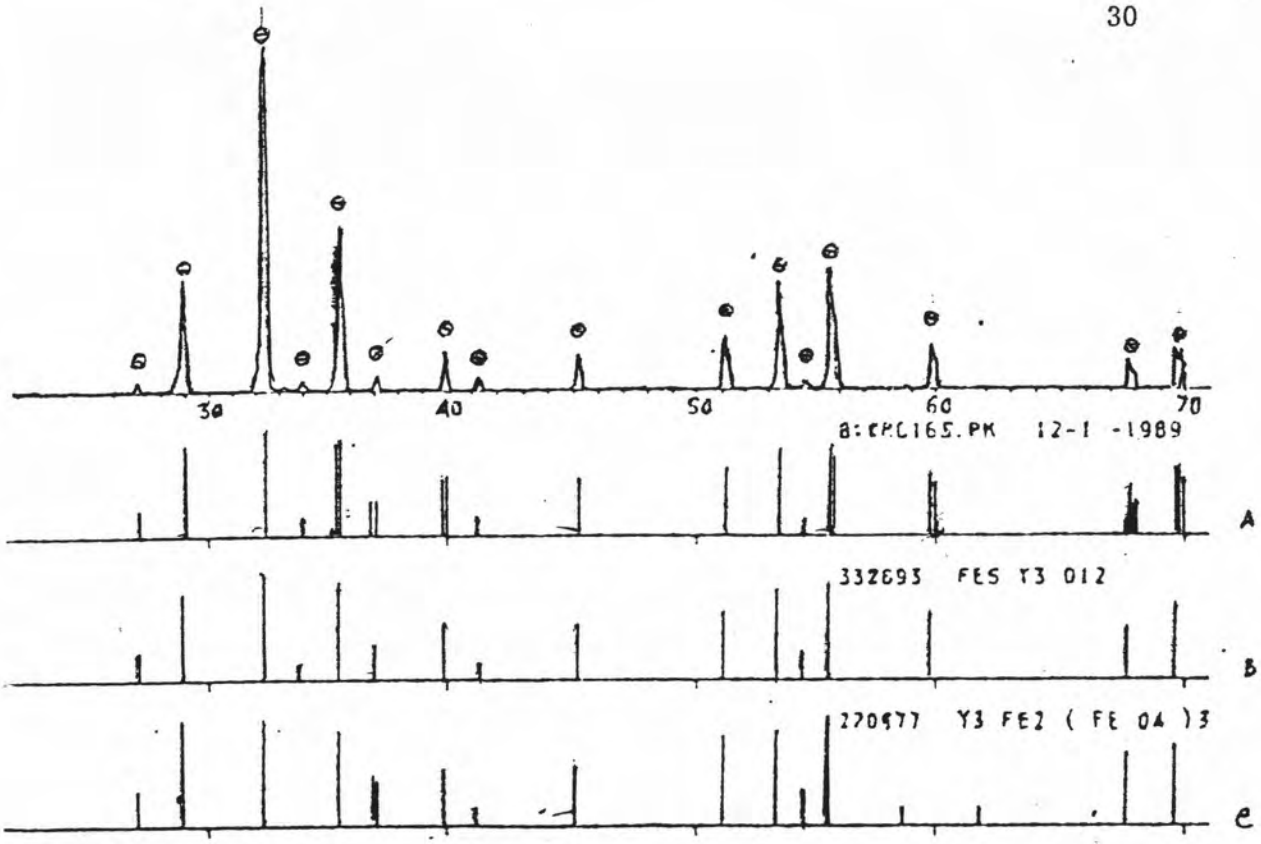
a. Ground the sample into fine powder and place it on a slide of dimension 1.5 cm * 1.5 cm. A drop of acetone is used to adhere the powder to the slide.

b. Place the slide in the X-ray diffractometer having a Cu target. Set the voltage to 40 keV, current to 30 mA and set the starting angle to 4 degrees and the stopping angle to 72 degrees. The wavelength of the X-ray is 1.5405 Å.

c. Calculate the d-spacing using the Bragg equation

$$2\,d\sin\theta = n\lambda$$

The x-ray pattern of our yttrium iron garnet taken with the X-ray diffractometer of the Geology Department is shown in Figure 3.6. The positions and intensities of the lines appearing in Figure 3.6 are listed in Table 3.2. Comparision of the positions of the lines with the positions of the yttrium iron garnet lines listed in the computer file on the diffractometer in the Scientific and Technological Research Equipment Centre points to our pellets being YIG.



Measure condition

Sample	Y3FE5012	
Target	Cu	
kV	45.0	kV
mA	30.0	mA
Start angle	5.00	deg.
Stop angle	70.00	deg.
Step angle	0.040	deg.
M. time	0.50	sec

Operator

Memo

2θ(°)	D(Å)	I(counts)	I/Io
17.56	5.046	137	8
26.96	3.304	56	3
28.88	3.089	603	34
32.36	2.764	1751	100
33.16	2.699	24	1
33.96	2.638	49	3
35.52	2.525	744	42
37.04	2.425	75	4
39.88	2.259	186	11
41.24	2.187	47	3
45.16	2.006	172	10
51.08	1.787	248	14
53.36	1.716	496	28
54.44	1.684	50	3
55.52	1.654	609	35
55.68	1.649	385	22
59.72	1.547	209	12
59.92	1.542	147	8
67.64	1.384	127	7
67.68	1.380	83	5
69.56	1.350	280	16
69.80	1.346	169	10

Figure 3.5 X-ray diffraction pattern of yttrium iron garnet and the results of a computer identification of the pattern. X-ray diffractometer of the Scientific and Technological Research Equipment Centre, Chulalongkorn University.

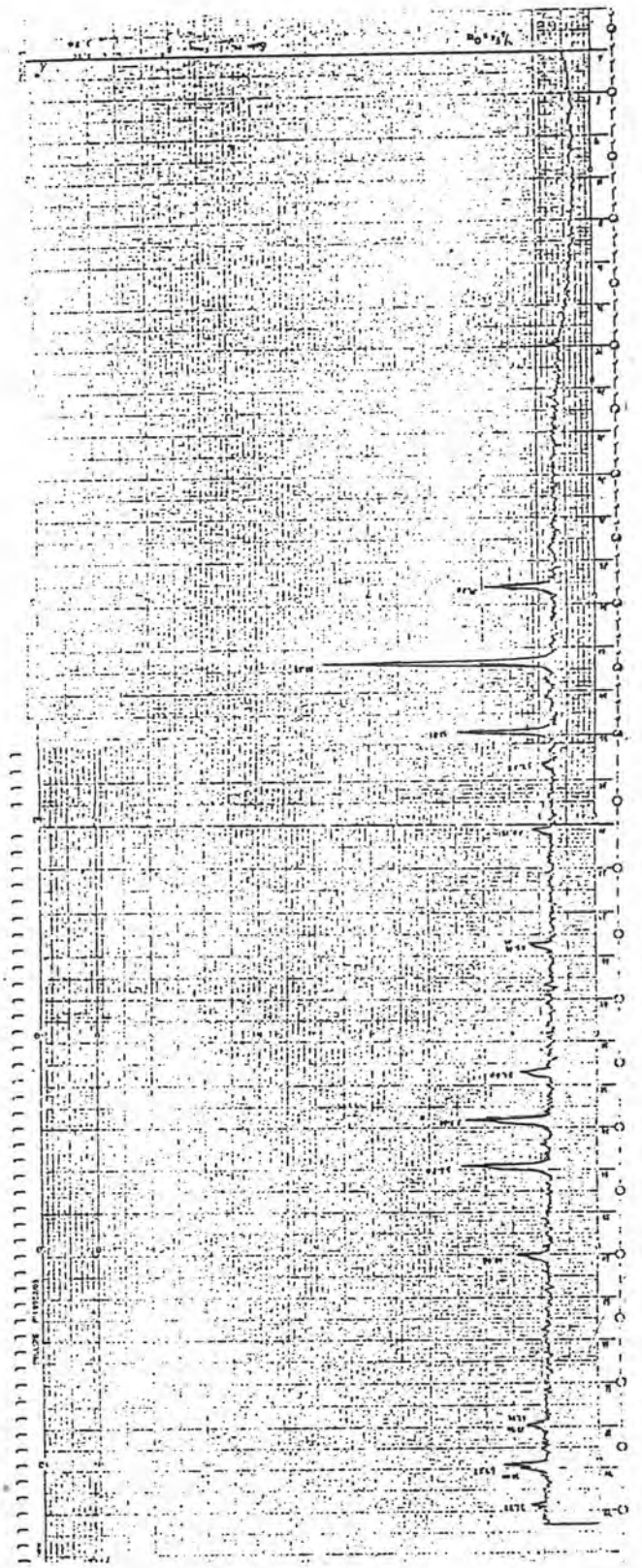


Figure 3.6 The X-ray diffraction pattern of our yttrium iron garnet .

Table 3.2

The positions and relative intensities of the lines appearing in Figure 3.6

2θ	sinθ	d Å	I/I_0
17.96	0.156	4.938	18
29.20	0.252	3.056	41
32.68	0.281	2.741	100
35.82	0.308	2.501	51
37.28	0.310	2.407	20
40.18	0.343	2.246	24
45.48	0.386	1.995	25
51.40	0.434	1.775	29
53.64	0.451	1.708	49
55.80	0.468	1.644	50
60.02	0.500	1.540	30
67.88	0.558	1.380	27
67.94	0.559	1.378	27
69.82	0.572	1.346	35
70.00	0.574	1.342	29
71.72	0.586	1.314	27