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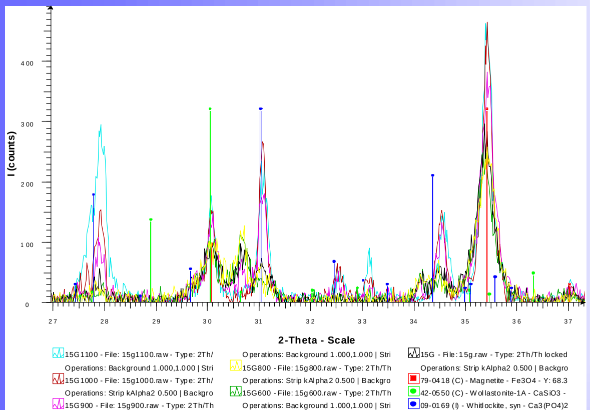
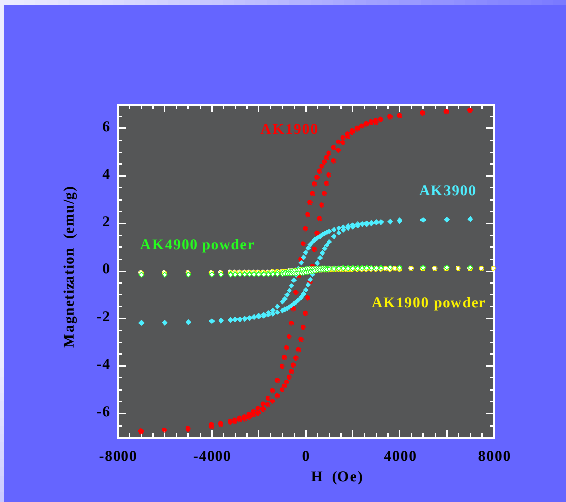
Processing and Magnetic Properties of BIOCERAMICS





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Goals of the project

Research the correlation between processing, magnetic and structural properties of bioactive, ferromagnetic glass-ceramics with base components

Fe_3O_4, CaO, P_2O_5, SiO_2, Na_2O

OUTLINE

- **About Bioactive Ferromagnetic Glass-Ceramics**
- **Sample preparation**
  X-Ray Characterization
- **Present Work: Magnetic Measurements at Oak Ridge National Lab**

A NEW TECHNIQUE

HYPERTHERMIA

❖What is HYPERTHERMIA?

❖**Hyperthermia is, very simply, the application of concentrated therapeutic heat to treat cancer.**

❖**Hyperthermia, as surgery, chemotherapy and radiation, is now recognized as the "fourth modality" in approved cancer treatment.**

❖**Many bone tumors require surgery, but new techniques are now available that allow patients with certain bone tumors to avoid surgery.**

Hyperthermia can be used for bone and soft tissue tumors treatment.

CMF is a cartilage tumor

❖Ferromagnetic Bio-Ceramics

❖Are bioactive materials developed from the original bioglass ceramics by L. Hench (1969) with the addition of Fe_2O_3 in the system P_2O_5, SiO_2, CaO.

❖Contain *magnetite* (Fe_3O_4) that generates heat when an *a.c. magnetic field* is applied, by hysteresis loss. Clinically tested for *hyperthermic treatment* of the bone cancer.

❖ Such application is based on the fact that cancer cells have a lesser surviving fraction on heat treatment than ordinary tissues at 42-45 ^oC .

♦ Biological tests

Rabbit tibia was packed with granules of this glass-ceramics and also ceramics pins was used as intramedullary fixation rods for rabbit tibiae.

This ceramics is considered capable of not only reinforcing the weakened bone, but also allowing hyperthermic treatment of the tumor us the figures shows.

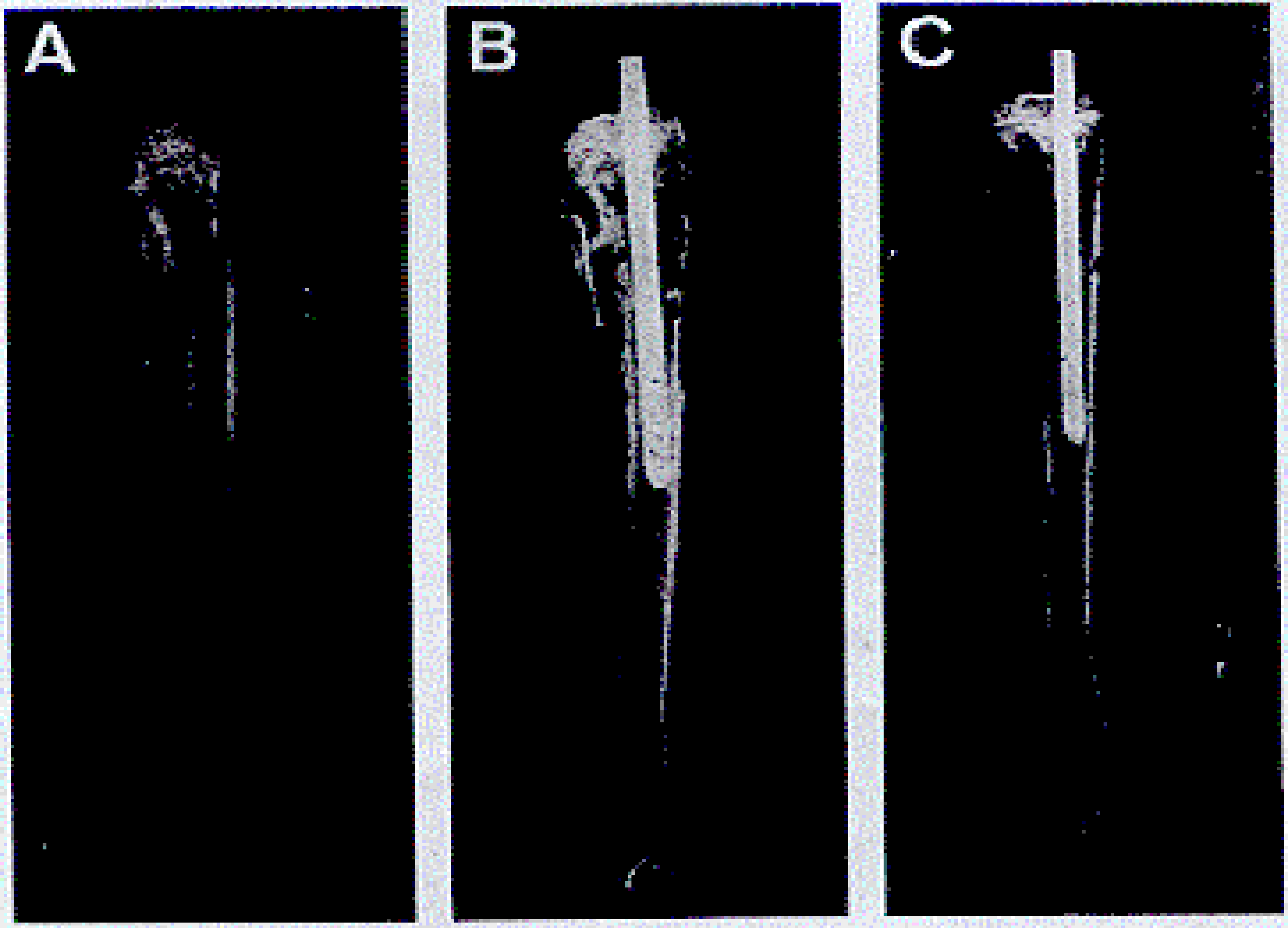


Figure 3-Soft X-ray photographs of a tibia.
A: No Treatment group, B: Pinning group, C: Pinning+HT (hyper-thermia) group.

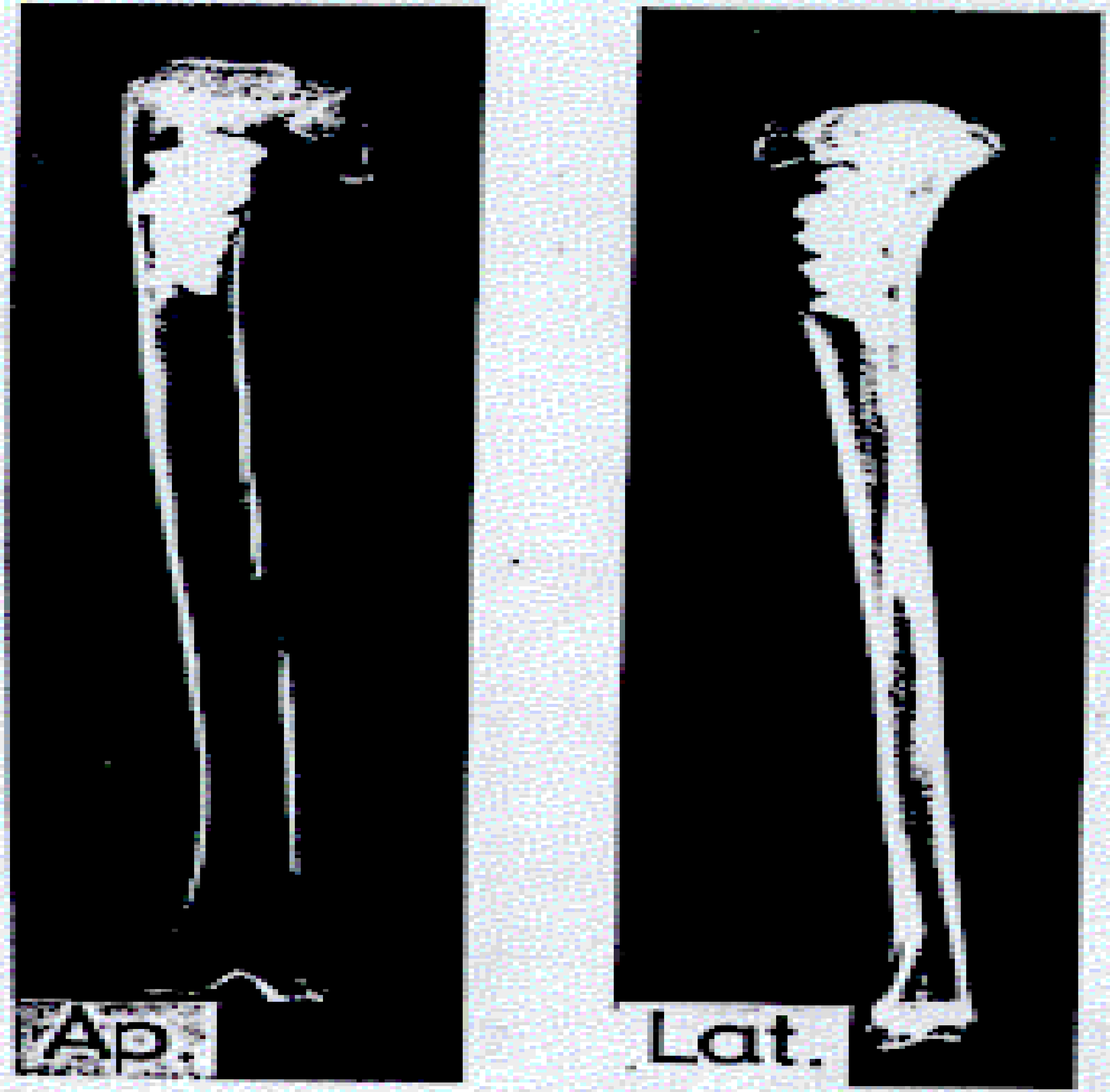


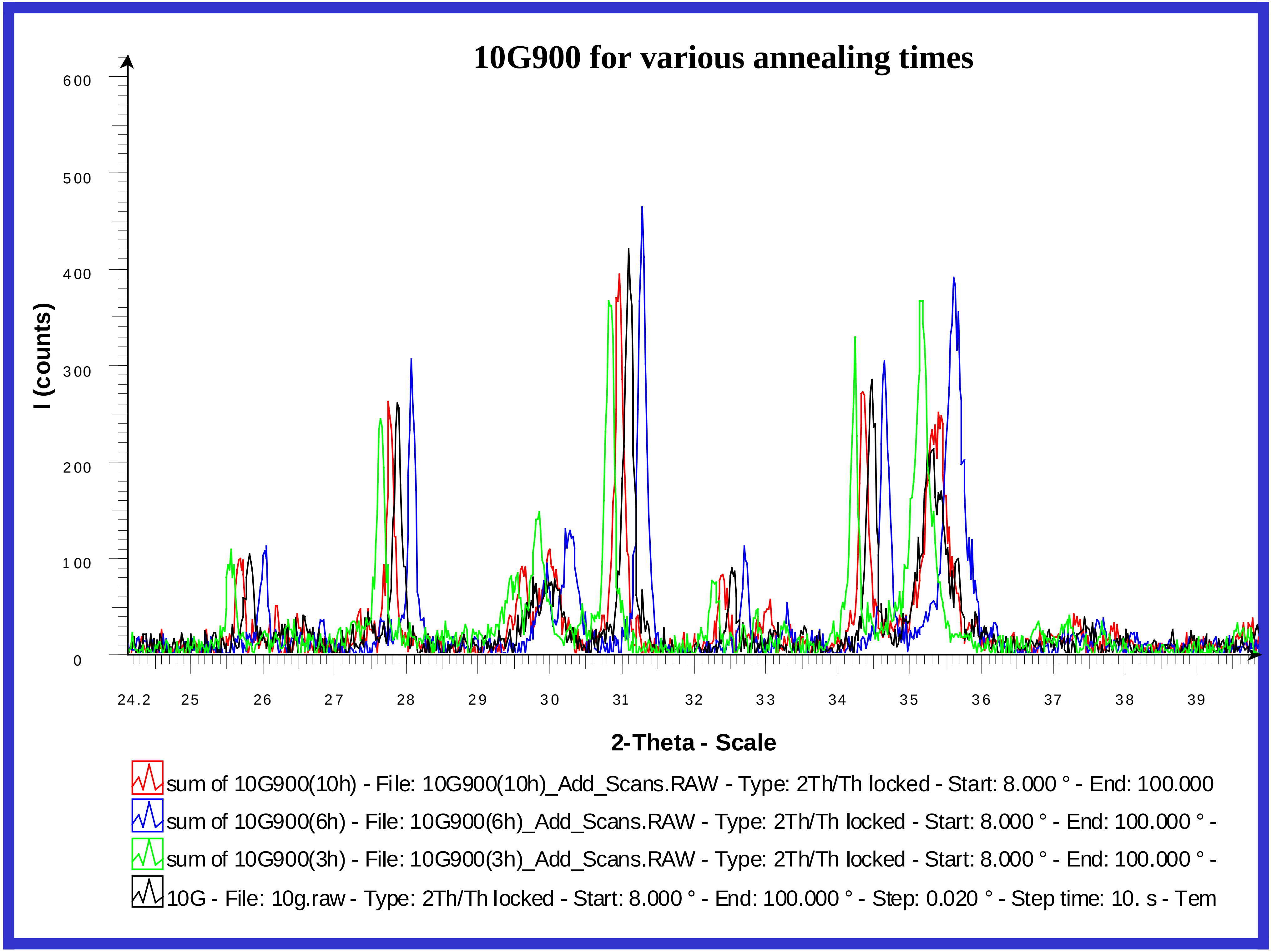
Figure 2. Rabbit tibia filled with granules of glass-ceramic in bone marrow.

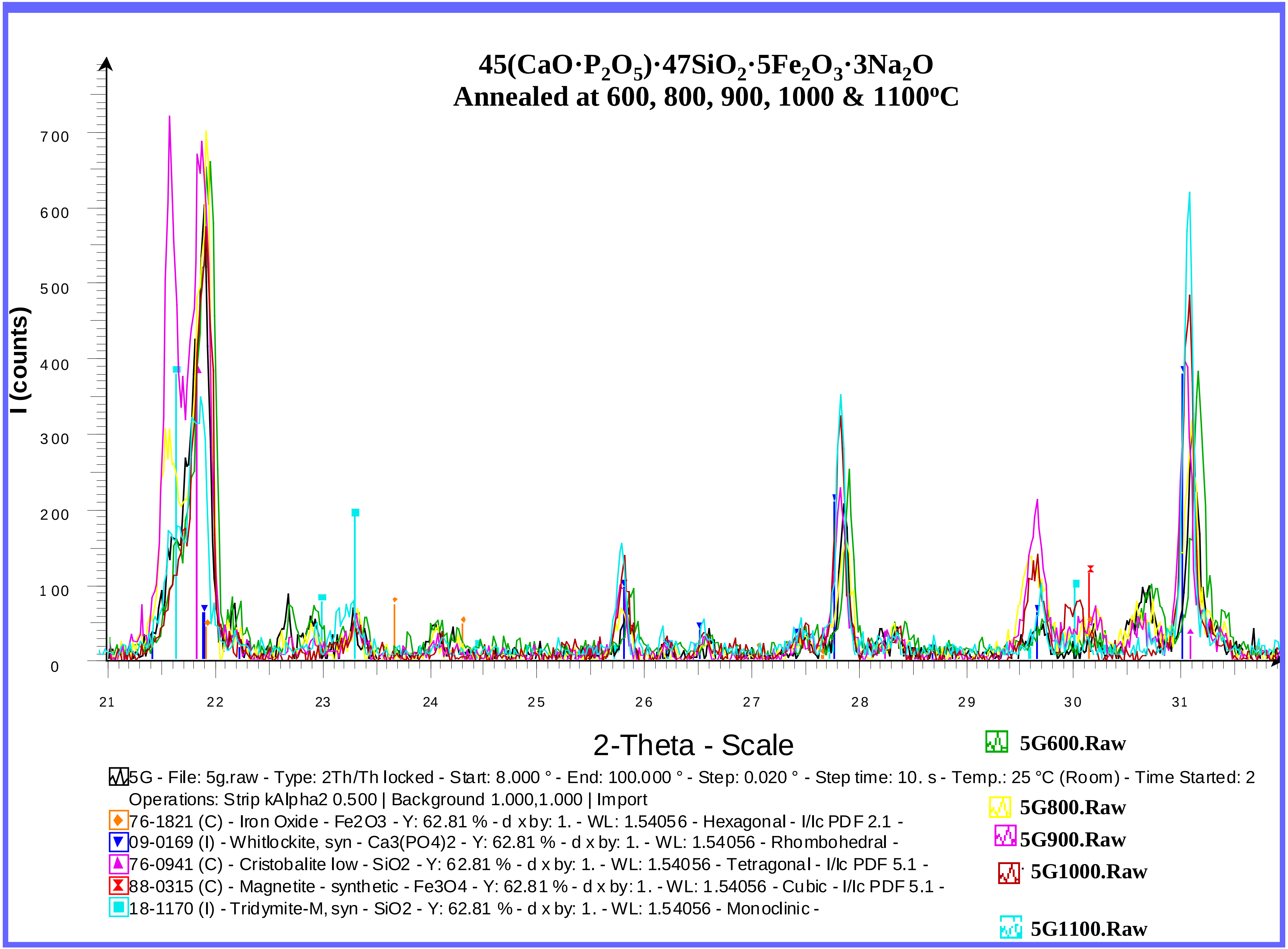
SAMPLES	STARTING COMPOSITION	PHASE IDENTIF.	Ca/P	MELT. TEMP (^{o}C)	ANNEALING TEMP(^{0}C) (6h)
5G	**45(CaO·P_2O_5)·47SiO_2·5Fe_2O_3·3Na_2O**	$Ca_3(PO_4)_2$ Fe_2O_3 SiO_2	1.67	1450	as prepared
5G600	"	$Ca_3(PO_4)_2$ Fe_2O_3 SiO_2	"	"	600
5G800	"	$Ca_3(PO_4)_2$ Fe_2O_3 SiO_2	"	"	800
5G900	"	$Ca_3(PO_4)$ Fe_2O_3 SiO_2 **Fe_3O_4**	"	"	900
5G1000	"	$Ca_3(PO_4)_2$ Fe_2O_3 SiO_2	"	"	1000
5G1100	"	$Ca_3(PO_4)_2$ Fe_2O_3 SiO_2	"	"	1100

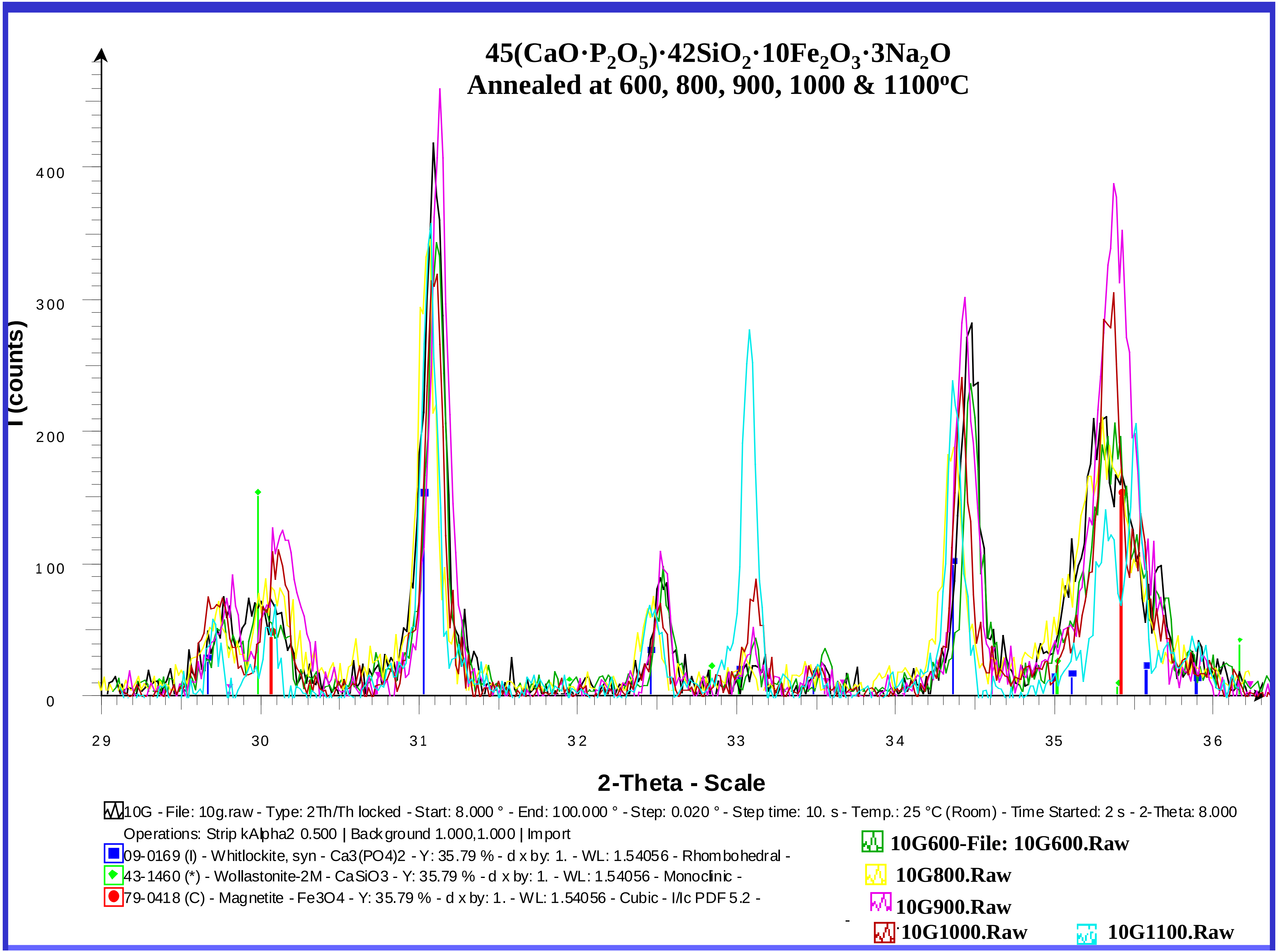
SAMPLES	STARTING COMPOSITION	PHASE IDENTIFIC.	Ca/P	MELT. TEMP (^{o}C)	ANNEAL. TEMP(^{0}C) (6h)
10G	**45(CaO·P$_2$O$_5$)·42SiO$_2$·10Fe$_2$O$_3$·3Na O**	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	1.67	1450	as prepared
10G600	"	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	"	"	600
10G800	"	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	"	"	800
10G900	"	$Ca_3(PO_4)$ $CaSiO_3$ **Fe_3O_4**	"	"	900
10G1000	"	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	"	"	1000
10G1100	"	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	"	"	1100

SAMPLES	STARTING COMPOSITION	PHASE IDENTIFIC	Ca/P	MELT. TEMP (ºC)	ANNEALING TEMP(ºC) /(6h)
15G	**45(CaO·P_2O_5)·37SiO_2 ·15Fe_2O_3 · 3Na_2O**	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	1.67	1450	as prepared
15G600	"	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	"	"	600
15G800	"	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	"	"	800
15G900	"	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	"	"	900
15G1000	"	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	"	"	1000
15G1100	"	$Ca_3(PO_4)_2$ $CaSiO_3$ Fe_2O_3 **Fe_3O_4**	"	"	1100

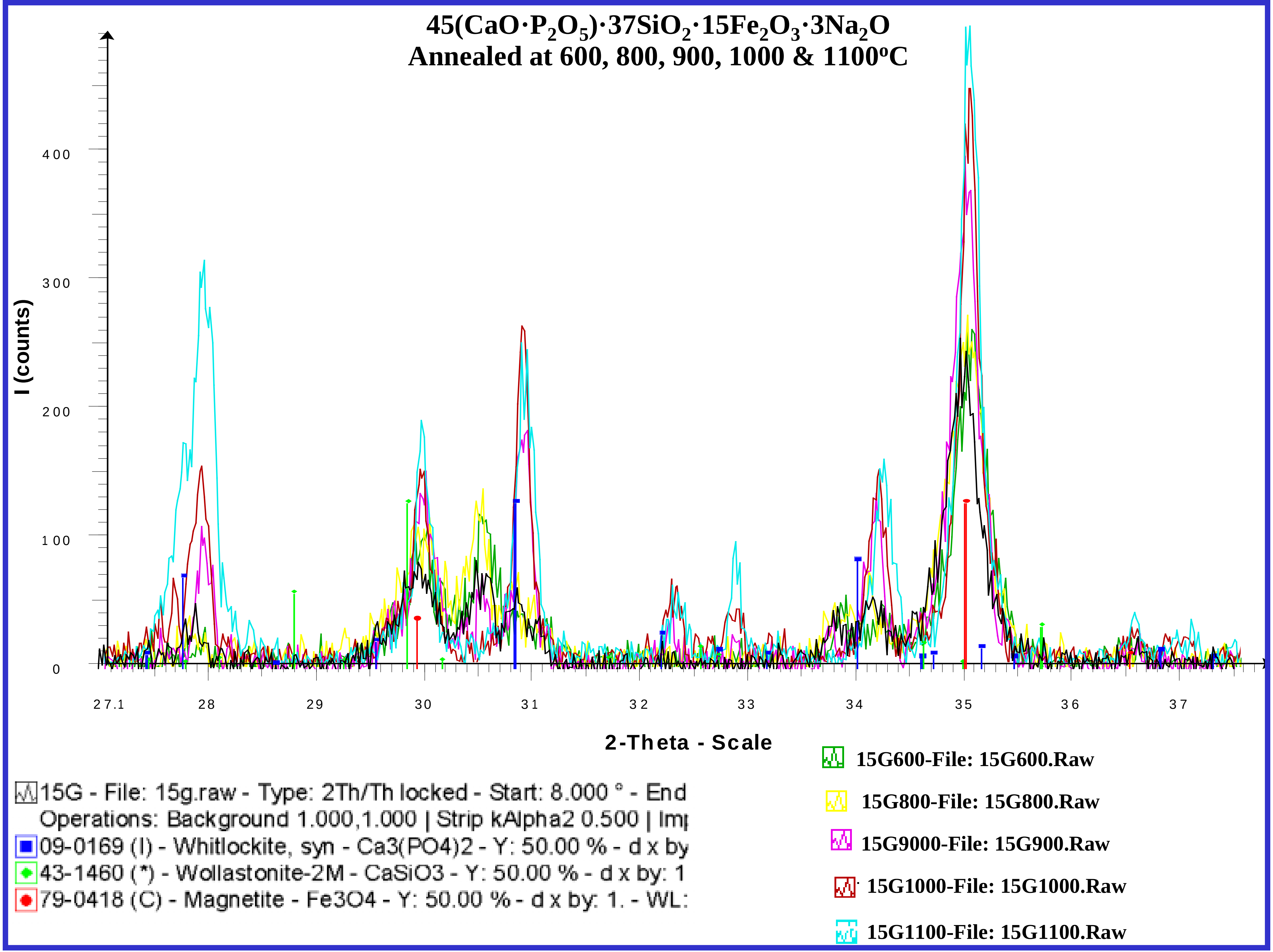
SAMPLES	STARTING COMPOSITION	PHASE IDENTIF.	Ca/P	MELT TEMP (^{o}C)	ANNEAL. TEMP(^{0}C) (6h)
20G	**$45(CaO \cdot P_2O_5) \cdot 32SiO_2 \cdot 20Fe_2O_3 \cdot 3Na_2O$**	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	1.6	1450	as prepared
20G600	"	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	"	"	600
20G800	"	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	"	"	800
20G900	"	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	"	"	900
20G1000	"	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	"	"	1000
20G1100	"	$Ca_3(PO_4)_2$ $CaSiO_3$ **Fe_3O_4**	"	"	1100

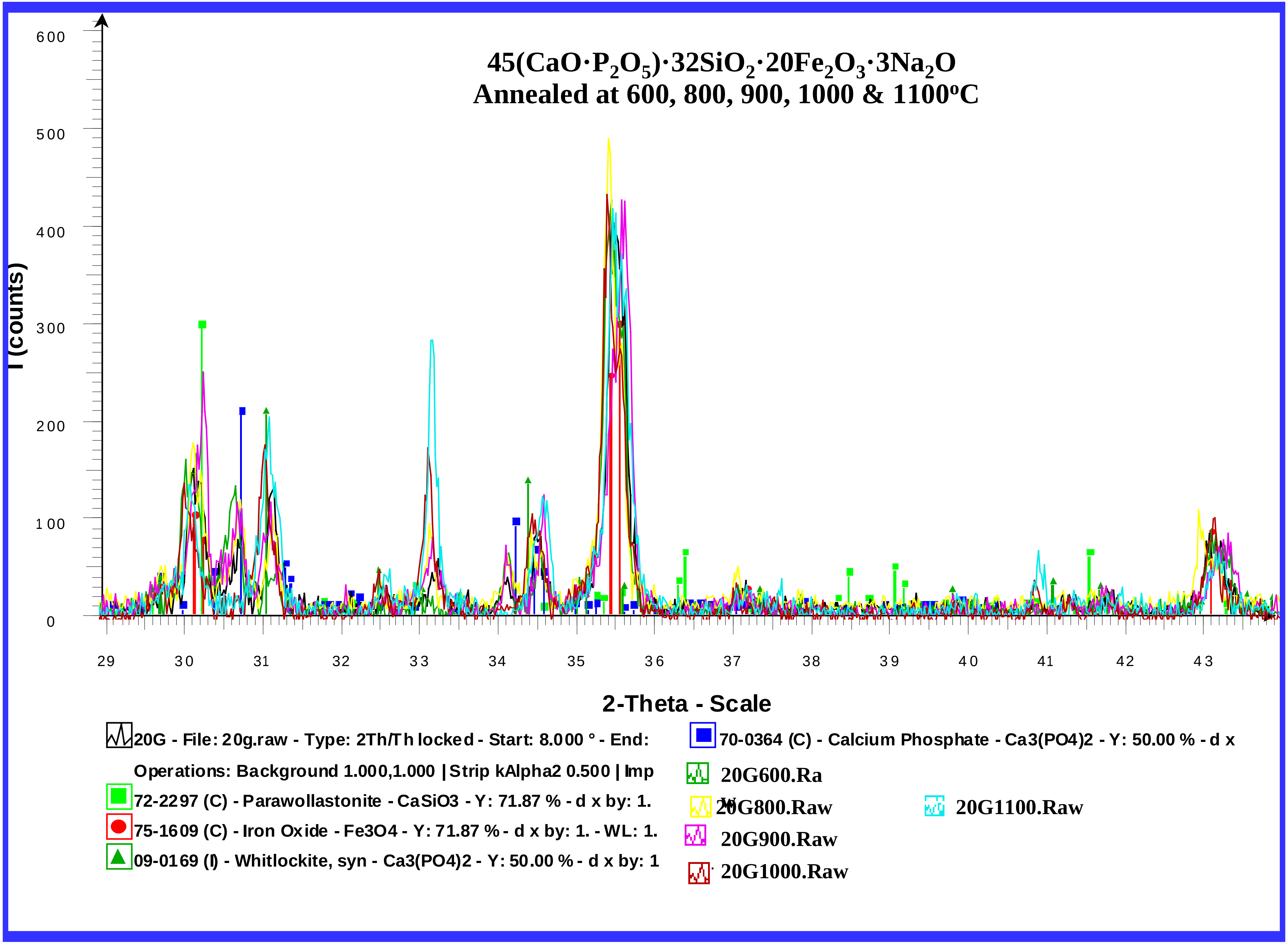

10G900 for various annealing times
I (counts)
600
500
400
300
200
100
0
24.2
25
26
27
28
29
30
31
32
33
34
35
36
37
38
39
2-Theta - Scale
sum of 10G900(10h) - File: 10G900(10h)_Add_Scans.RAW - Type: 2Th/Th locked - Start: 8.000 ° - End: 100.000
sum of 10G900(6h) - File: 10G900(6h)_Add_Scans.RAW - Type: 2Th/Th locked - Start: 8.000 ° - End: 100.000 ° -
sum of 10G900(3h) - File: 10G900(3h)_Add_Scans.RAW - Type: 2Th/Th locked - Start: 8.000 ° - End: 100.000 ° -
10G - File: 10g.raw - Type: 2Th/Th locked - Start: 8.000 ° - End: 100.000 ° - Step: 0.020 ° - Step time: 10. s - Tem


45(CaO·P2O5)·47SiO2·5Fe2O3·3Na2O
Annealed at 600, 800, 900, 1000 & 1100ºC
I (counts)
700
600
500
400
300
200
100
0
21
22
23
24
25
26
27
28
29
30
31
2-Theta - Scale
5G600.Raw
5G800.Raw
5G900.Raw
5G1000.Raw
5G1100.Raw
5G - File: 5g.raw - Type: 2Th/Th locked - Start: 8.000 ° - End: 100.000 ° - Step: 0.020 ° - Step time: 10. s - Temp.: 25 °C (Room) - Time Started: 2
Operations: Strip kAlpha2 0.500 | Background 1.000,1.000 | Import
76-1821 (C) - Iron Oxide - Fe2O3 - Y: 62.81 % - d x by: 1. - WL: 1.54056 - Hexagonal - I/Ic PDF 2.1 -
09-0169 (I) - Whitlockite, syn - Ca3(PO4)2 - Y: 62.81 % - d x by: 1. - WL: 1.54056 - Rhombohedral -
76-0941 (C) - Cristobalite low - SiO2 - Y: 62.81 % - d x by: 1. - WL: 1.54056 - Tetragonal - I/Ic PDF 5.1 -
88-0315 (C) - Magnetite - synthetic - Fe3O4 - Y: 62.81 % - d x by: 1. - WL: 1.54056 - Cubic - I/Ic PDF 5.1 -
18-1170 (I) - Tridymite-M, syn - SiO2 - Y: 62.81 % - d x by: 1. - WL: 1.54056 - Monoclinic -



45(CaO·P2O5)·42SiO2·10Fe2O3·3Na2O
Annealed at 600, 800, 900, 1000 & 1100ºC
I (counts)
0
100
200
300
400
29
30
31
32
33
34
35
36
2-Theta - Scale
10G - File: 10g.raw - Type: 2Th/Th locked - Start: 8.000 ° - End: 100.000 ° - Step: 0.020 ° - Step time: 10. s - Temp.: 25 °C (Room) - Time Started: 2 s - 2-Theta: 8.000
Operations: Strip kAlpha2 0.500 | Background 1.000,1.000 | Import
09-0169 (I) - Whitlockite, syn - Ca3(PO4)2 - Y: 35.79 % - d x by: 1. - WL: 1.54056 - Rhombohedral -
43-1460 (*) - Wollastonite-2M - CaSiO3 - Y: 35.79 % - d x by: 1. - WL: 1.54056 - Monoclinic -
79-0418 (C) - Magnetite - Fe3O4 - Y: 35.79 % - d x by: 1. - WL: 1.54056 - Cubic - I/Ic PDF 5.2 -
10G600-File: 10G600.Raw
10G800.Raw
10G900.Raw
10G1000.Raw
10G1100.Raw


45(CaO·P2O5)·37SiO2·15Fe2O3·3Na2O
Annealed at 600, 800, 900, 1000 & 1100ºC
I (counts)
400
300
200
100
0
27.1
28
29
30
31
32
33
34
35
36
37
2-Theta - Scale
15G - File: 15g.raw - Type: 2Th/Th locked - Start: 8.000 ° - End
Operations: Background 1.000,1.000 | Strip kAlpha2 0.500 | Im
09-0169 (I) - Whitlockite, syn - Ca3(PO4)2 - Y: 50.00 % - d x by
43-1460 (*) - Wollastonite-2M - CaSiO3 - Y: 50.00 % - d x by: 1
79-0418 (C) - Magnetite - Fe3O4 - Y: 50.00 % - d x by: 1. - WL:
15G600-File: 15G600.Raw
15G800-File: 15G800.Raw
15G9000-File: 15G900.Raw
15G1000-File: 15G1000.Raw
15G1100-File: 15G1100.Raw



45(CaO·P2O5)·32SiO2·20Fe2O3·3Na2O
Annealed at 600, 800, 900, 1000 & 1100ºC
I (counts)
600
500
400
300
200
100
0
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
2-Theta - Scale
20G - File: 20g.raw - Type: 2Th/Th locked - Start: 8.000 ° - End:
Operations: Background 1.000,1.000 | Strip kAlpha2 0.500 | Imp
72-2297 (C) - Parawollastonite - CaSiO3 - Y: 71.87 % - d x by: 1.
75-1609 (C) - Iron Oxide - Fe3O4 - Y: 71.87 % - d x by: 1. - WL: 1.
09-0169 (I) - Whitlockite, syn - Ca3(PO4)2 - Y: 50.00 % - d x by: 1
70-0364 (C) - Calcium Phosphate - Ca3(PO4)2 - Y: 50.00 % - d x
20G600.Ra
20G800.Raw
20G1100.Raw
20G900.Raw
20G1000.Raw

PRESENT WORK

We are investigating correlation between processing parameters (starting composition, heat treatment)

and

magnetic properties of ferromagnetic bioceramics.

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FUTURE WORK

- Quantitative analysis of the phases using the Rietveld method

- Magnetic structure from neutron powder diffraction

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ACKNOWLEDGEMENTS

We gratefully acknowledge SURA for supporting our project.

Special thanks to our collaborators in the magnetic Measurements Laboratory of the ORNL for their help and support.

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