Preparation of Apatite-Wollastonite and Phlogophite Glass Ceramic and Study of Its Properties

A THESIS SUBMITTED IN PARTIAL FULLFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF BACHELOR OF TECHNOLOGY

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CERTIFICATE

This is to certify that the thesis entitled, ***"Preparation of Apatite-Wollostonite And Phlogophite Glass Ceramic and study of its properties"*** submitted by ***Mr. Anuj Kumar*** in partial fulfillment of the requirements of the award of Bachelor of Technology Degree in Ceramic Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by him under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other university / institute for the award of any Degree.

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ABSTRACT

Since the discovery of bioglass by Hench in 1969, there has been a new beginning of making biocompatible materials having improved properties. The major success was achieved by kokubu with the formation of AW glass. Since then a lot of research has been done on AW glass to improve its properties.

Mica crystals possess excellent cleavage planes perpendicular to the c-axis because of weak bonding of alkali layers and hence have excellent machinability which can be utilized to prepare machinable bioactive glass.

In the current project work different proportion of Phlogophite(10 – 50%) were mixed with apatite-wollastonite glass ceramic and to determine its machinability and properties. From mixture of AW and Ph pallets were prepared and were crystallized at 950°C. X – Ray diffraction analysis of the samples has confirmed the desired phase formation o f crystals of AW and Ph.

The apparent porosity increases and bulk density decreases with the increase in Ph phase. Also the hardness decreases with the incorporation of Ph, hence suggesting that the machinability is increasing. The drilling operation done on two of the samples having 60:40 and 50:50 composition shows machinability. But both the samples break at the last stage of drilling which may be due to uneven phase distribution of Ph phase.

CHAPTER 1

INTRODUCTION

With the advent of modern era and with the development of medical science the living of people around the globe has changed a lot. Bioceramics has a major role to play in the field of repair and reconstruction of diseased, damaged or worn out parts of the body. Bioceramics are produced in a variety of forms and phases and serve many different functions of the body. The bioactive glass was first produced by Hench et al in 1969 which can bond chemically to the bone. Since then a lot of research has undergone for the development of biomaterials. [1]

The main achievements in glass production is the design of bioactive glass materials and a lot of research is being carried out around the globe for improving its properties to make it as biocompatible as possible. To make the material biocompatible so that it can interact with the living tissue there should be formation of apatite phase which is a part of natural bone and provides strength and growth ability. [2 - 4]

The A/W glass–ceramic studied by Kokubo in 1980 shows high bioactivity and high mechanical strength compared to other glasses and glass–ceramics which is due to the relatively high fracture toughness due to the precipitation of β-wollastonite in addition to apatite. Crystallisation of the parent glass in a bulk form led to the occurrence of large cracks in the crystallised sample while crystallisation of the same glass in a powder compact led to the formation of a crack-free dense crystallised sample due to uniform crystallization of apatite and wollastonite as demonstrated by . Kokubo et al. [5,6]

A/W glass–ceramics are one of the most bioactive materials having bioactivity index slightly higher than that of dense sintered hydroxyapatite and requires less time for bone bonding in comparison with hydroxyapatite.[5]

For the improved mechanical properties and machineability controlled crystallization of both mica and apatite can be carried out. Desired bioactivity is taken care by apatite formation and machinability by mica phase. The special characteristic of the glass-ceramic was that two distinct crystal phases were precipitated in parallel: apatite and mica were produced simultaneously with a volume nucleation mechanism. The resultant glass ceramic gives high value of mechanical properties. [7]

Fluormica glass–ceramics shows a favorable combination of thermal, electrical and biomedical properties which can be easily cut, drilled and turned with conventional tools. Laminated structure of mica crystals is, directly responsible for the desirable machinability because they cleave easily along the interfaces between layers while being machined. [8 - 10]

Machineability of a glass ceramic is appropriate if it can be turned, milled, drilled and tapped without breaking as normal ceramics would. Beall et al started the development of such materials around 1970.The controlled cryslallization of apatite and phlogophite phase can produce the desired property of bioactivity and machineability both at a time. [8]

In the current research work Apatite-Wolastonite and Phlogophite powders were prepared separately and mixed in different composition and were fired at 950°C crystalline phase of both A/W and Ph had formed as confirmed by the XRD analysis and different mechanical properties of them have been studied.

CHAPTER 2

LITERATURE REVIEW

Apatite-wollastonite glass-ceramic is a matrix of small apatite particles effectively reinforced by wollastonite phase. A-W glass-ceramic has higher value of the bending strength, fracture toughness and Young's modulus among bioactive glass and glass ceramics which makes it useful in major compression load bearing applications. [11]

Controlled crystallization gives apatite phase where a melt of variable concentration could be separated into mica and apatite crystals. Fluorapatite is a kind of hydroxyapatite where the hydroxylic groups (OH^{-1}) are substituted by F^{-1} ions which results in reduction of the solubility, starts the formation of bone tissue with increases in the compression strength. The extraction of ions in the surrounding bone forms stronger bonds since it strengthens the tissue next to the implanted one. B.I. Bogdanov et al prepared three kinds of glasses by using system SiO_2–Al_2O_3–P_2O_5–CaO–CaF_2 and determined the conditions for crystallization in order to synthesize glass ceramic materials. Bioactive glass ceramic materials were prepared by thermal treatment of the glass mixture-melting at 1450°C for 2h and crystallization at 680°C for 12 h. [2]

High mechanical strength is produced between bone and glass ceramic owing to strong chemical bond among them. Crystals such as apatite, wollastonite and whitlockite precipitate from the glass matrix after heat treatment which enhances the mechanical strength and promote the bioactivity of the glass ceramic. The types of crystals formed in the glass matrix depends on the heat treatment and glass composition.C.K. Chang et al found that a dense material can be obtained by sintering of A/W glass ceramics at 1200°C. The main crystal phase identified were Whitlockite and wollastonite having different morphologies in the material with a glass matrix found on the boundaries. The crystals were non-stoichiometic with Si in whitlockite and Mg in wollastonite. Silicon in whitlockite increases the lattice volume while Mg in wollastonite did not influence the lattice parameters. Al_2O_3 introduced by milling remained in the glass matrix and had no effect on the crystal phases. [12]

Machinable glass–ceramics are based on phlogopite. A. Faeghi-Nia et al studied the crystallization behavior of a glass–ceramic based on $KMg_3AlSi_3O_{10}F_2$ micas by varying the B_2O_3 content in glass composition. By Increasing the B_2O_3 content the glass transition temperature, dilatometric softeningpoint and first peak crystallization temperature decreases. [13]

S. Habelitz et al studied the machinability of mica glass ceramics which exhibits high thermal shock resistance and fracture toughness but have low mechanical strength. Structure and morphology of the crystal mainly determine these properties. Mica crystals possess excellent cleavage planes perpendicular to the c-axis because of weak bonding of alkali layers. [14]

According to Wolfram Hioland, machinable glass-ceramics can be developed with flat mica crystals of phlogopite-type. Phlogopite crystal was formed with controlled crystallization producing a new machinable glass-ceramic. Having proven biocompatibility and the favorable mechanical processing this glass-ceramic material has been used in head and neck surgery. Controlled crystallization of both mica and apatite yield bioactive glass ceramic. The required bioactivity was produced using a minimum $A1_20_3$ content as in the composition 38.7 wt% SiO_2, 1.4$A1_2O_3$, 27.7MgO, 10.4 CaO, 6.8K_2O, 4.9 F, 8.2P_2O_5, 1.9TiO_2. [7]

Tadashi Kokubo found that crystalline phases of apatite, $Ca_{10}(PO_4)_6O,F_2)$ and β-wollastonite is found on the controlled heat treatment of an MgO-CaO-SiO_2-P_2O_5 glass in MgO-CaO-SiO_2 glassy matrix which showed bioactivity and a fairly high mechanical strength which decreased only slowly, even under load-bearing conditions in the body and can be clinically as artificial vertebrae, iliac bones, etc. Apatite layer formation on its surface in the body is the reason for its bioactivity which is formed by dissolution of calcium and silicate ions from the glass ceramic.

Kokubu et al synthesize a microcomposite as like bone which an assembly of apatite small particles is effectively reinforced by p-wollastonite ($CaO.SiO_2$) having a silicate chain structure, by crystallization of an MgO-CaO-SiO_2-P_2O_5, glass. Bone like apatite layer is formed on their surfaces which is responsible for bonding with living bone. [15]

LI Rounan et al states that a phlogophite glass ceramic contains crystalline phase of fluorophlogophite which does not only have good dielectric properties, excellent thermal shock resistance and good mechanical strength but also very good machinabilities which can be machined to precise tolerances. These machinable properties of phlogopite-based glass ceramics having bioactivity due to apatite phase broaden the application of glass in medical field. [16]

Aida Faeghi-Nia et al states that most studied bio glass ceramic are A/W glass ceramic and mica based glass ceramic. The needle shaped wollastonite crystallite imparts high strength and fracture toughness to final glass ceramic. The phlogopite (Ph) type materials contain a significant amount of alkali metal ions, which results degradation of mechanical properties in living system. For obtaining sufficient bioactivity the strength and machinability of these materials is always compromised. The properties of glass–ceramics are mainly determined by the amount and the distribution of the crystalline phases depends on the original glass composition. [17]

CHAPTER 3

Apatite Formation at the Surface of the AW Glass Ceramic

When AW glass ceramic is immersed in simulated body fluid then the concentration of elements present in it changes. AW dissolve appreciable amount of calcium and silicate ions. There is decrease in phosphorus concentration with immersion of AW due to formation of apatite on its surface hence consuming phosphate ion from the fluid. Hence there is dissolution of calcium and/or silicate ion which plays an important role in the formation of apatite layer. [18]

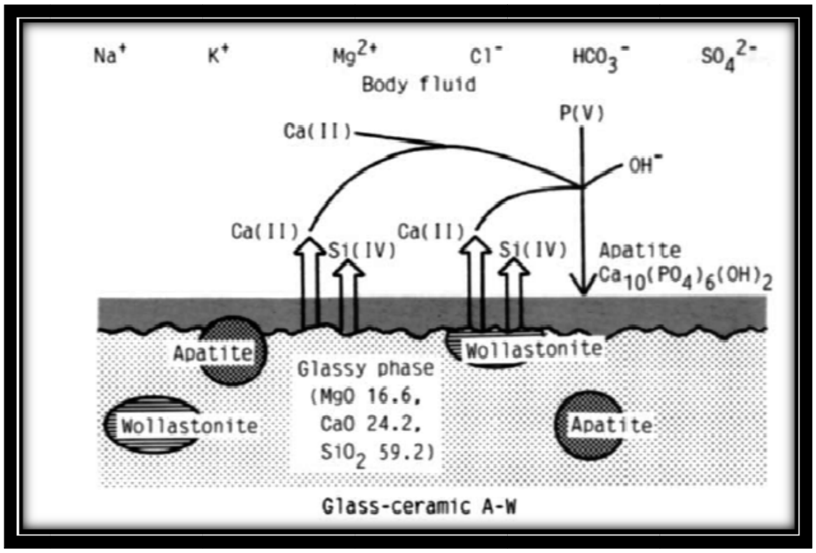

Fig1. Schematic representation of reaction at the surface of glass ceramic A-W in the body [15]

The apatite present in the glass ceramic does not dissolve any ions, since the body fluid is already supersaturated for the apatite34. The phosphate ions required for formation of apatite are only supplied from the surrounding body fluid. In this way apatite layer is formed in the surface of AW glass ceramic in the body. [15]

Machineability of Mica Glass Ceramic

Mica glass ceramics exhibit excellent machineability which depends on the structure and morphology of the mica crystals. Fig. 1 shows the structure of common fluorophlogopite ($K/NaMg_3AlSi_3O_{10}F_2$) mica. The (001)-plane of phlogopite is composed of a two-dimensional network forming hexagonal rings. A layer of magnesium-cations in octahedral coordination is located between two tetrahedral layers. The resulting triple layers are connected to each other by relatively large alkali ions, e.g., K^+ or Na^+. Due to presence of weak bonding of alkali layers mica crystals possess excellent cleavage planes perpendicular to the c-axis. Across the basal plane fracture propagation of mica is more difficult which induces fracture to follow cleavage planes or mica glass interfaces. Since the crystals are in mutual contact, a series of deflections, branching and often blunting of cracks absorbs energy.

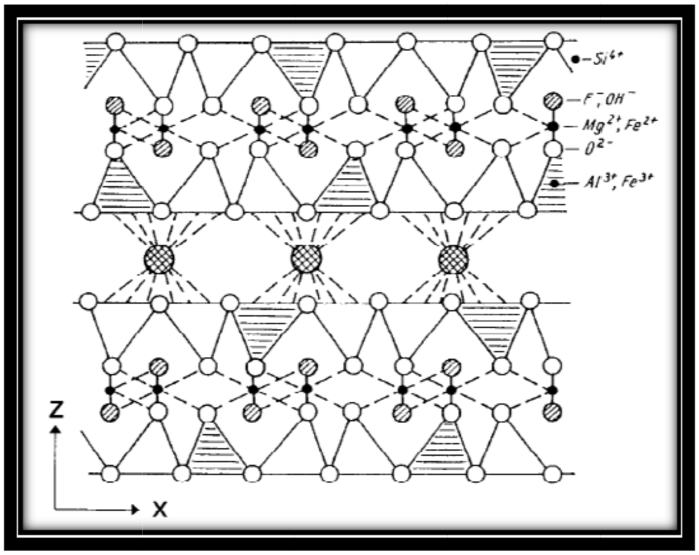

Fig. 2. Crystal structure of the (010)-plane of fluorophlogopite ($K/NaMg_3\,AlSi_3O_{10}\,F_2$)[14]

During machining fractures are deflected and enclosed glassy regions are dislodged. The material can thus be machined without shattering. Hence it is highly machinable.[20]

CHAPTER 4

4.1 Plan of the work

The present project work is to be carried out according to the following plan:

1. The objective of the project is to prepare Apatite – Wollastonite and Phlogophite glass ceramic and study it properties.
2. Apatite – Wolastonite frits is to be prepared using reagents grades $(NH_4)HPO_4$, $CaCO_3$, CaF_2, $MgCO_3$ and pure SiO_2 sand by melting at 1450°C and quenching in cold water.
3. Phlogophite frits is to be prepared using reagents grades grades K_2CO_3, $MgCO_3$, MgF_2, Al_2O_3 and pure SiO_2 sand by melting at 1500°C and quenching in cool water.
4. Both AW and Ph frits will be ball milled using zirconia balls and the powders will be screened to less then 45µm size.
5. Different proportion of the AW and Ph powders will be mixed and to prepare pallets.
6. The different proportions are 100% AW, 90% AW – 10% Ph, 80% AW – 20% Ph, 70% AW – 30% Ph, 60% AW – 40% Ph, 50% AW – 50% Ph.
7. The pallet of 100% AW will be fired at 900°C and other pallets will be fired at 950°C.
8. X – Ray diffraction will be done for all the sample to identify the phases formed.
9. The apparent porosity and bulk density of the samples is to be found out.
10. Hardness of the samples is to be tested.
11. Samples will be drilled to check out the machinability of the samples.

4.2 Process Flow Chart

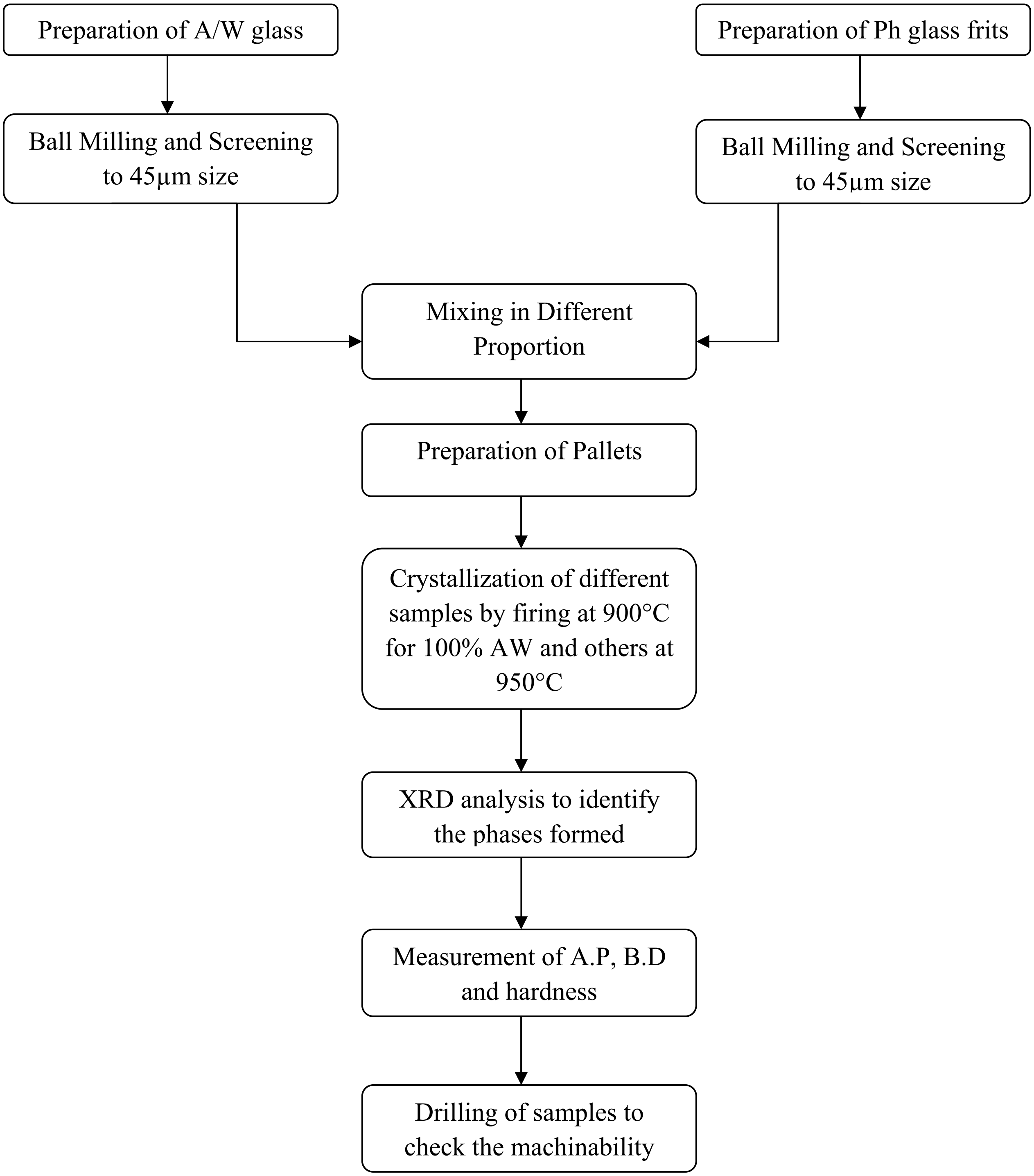

Fig 3. Process Flow Chart

CHAPTER 5

Experimental Procedure

5.1 Apatite Wollastonite glass Preparation

Tha Apatite – Wollastonite (A/W) glass frits were prepared from reagents grades $(NH_4)HPO_4$, $CaCO_3$, CaF_2, $MgCO_3$ and pure SiO_2 sand. Table1 gives the composition of batch.

Table 1. Composition of Apatite – Wollastonite glass.

S.No	Material	Wt(%)	Material Used	Wt(gm)
1	SiO_2	34.2	SiO_2	17.1000
2	P_2O_5	16.3	$(NH_4)_2HPO_4$	15.1520
3	CaO	45	$CaCO_3$	40.1768
4	F	0.5	CaF_2	0.5132
5	MgO	4	$MgCO_3$	4.2000

The above materials were taken in required amount and mixed properly. The composition was chosen from the Kokubo et al [1] work. The glass batch was calcined for 1hr at 900°C and then melted at 1450°C in raising hearth furnace in sillimanite crucible. The heating rate was kept at 3°C/min. The melting time was 1h and melts were poured into cold water to obtain glass frits. Then the frits were kept for drying in hot air oven at 110°C. After drying the frits were grounded using cylindrical zirconia balls by using pot mill. After milling the powder was screened to less then 45µm.

5.2 Phlogophite Glass Preparation

The Phlogophite (Ph) glass frits were prepared from reagents grades K_2CO_3, $MgCO_3$, MgF_2, Al_2O_3 and pure SiO_2 sand. Table 2 gives the composition of batch.

Table 2. Composition of Phlogophite glass.

S.No	Material	Wt(%)	Material Used	Wt(gm)
1	K_2O	8.188	K_2CO_3	6.0103
2	MgO	20.906	$MgCO_3$	21.9513
3	Al_2O_3	26.655	Al_2O_3	13.3275
4	SiO_2	33.449	SiO_2	16.7245
5	MgF_2	10.801	MgF_2	5.4005

The above materials were taken in required amount and mixed properly. 17.1652gm of H_3BO_3 was added to in the above composition and mixed properly. The composition was chosen from the Aida Faeghi-Nia et al.'s[17] work. The glass batch was calcined for 1hr at 900°C and then melted at 1500°C in raising hearth furnace in sillimanite crucible. The heating rate was kept at 3°C/min. The melting time was 1h and melts were poured into cold water to obtain glass frits. Then the frits were kept for drying in hot air oven at 110°C. After drying the frits were grounded using cylindrical zirconia balls by using pot mill. After milling the powder was screened to less then 45µm.

5.3 Flow Chart of Glass Powder Preparation

Apatite – Wollastonite

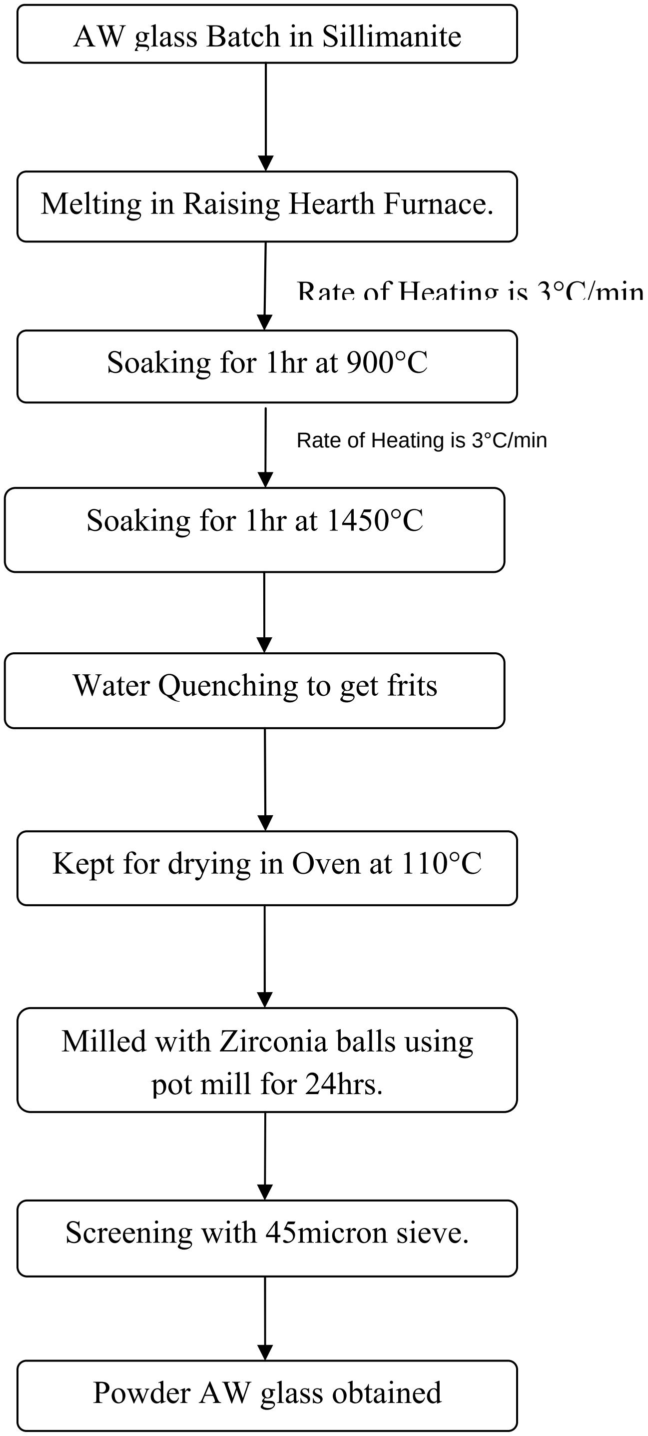

Fig 4. Flow Chart for Preparation of AW Powder

Phlogophite Glass

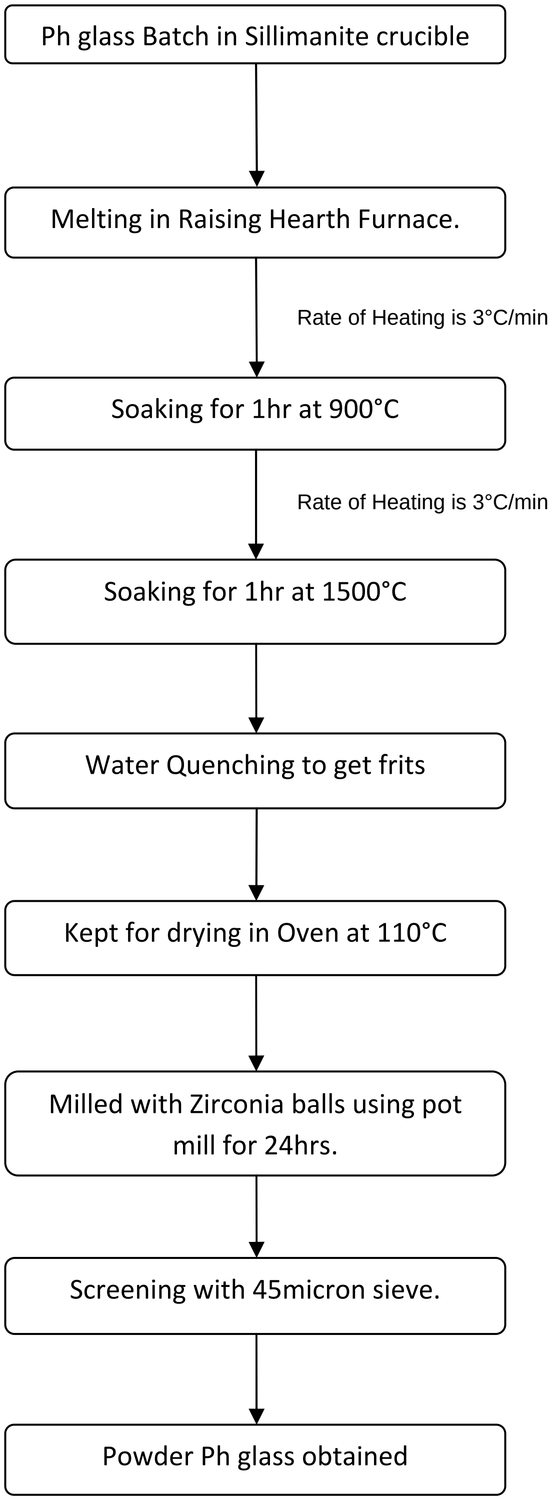

Fig 5. Flow Chart for Preparation of Ph Powder

5.4 Apatite –Wollastonite and phlogophite Glass ceramic:

The matrix studied in this project was A/W frit to which Ph glass was added in different ratio varying from 10% Ph to 50% Ph. The powder were mixed using agate motor and pallets were prepared using die.. The load given was 4ton with 1 min of dwell time. The binder used was PVA. The pressing machine used was Carver Press. Table 3 shows the composition of frit mixtures.

Table 3. Composition of frit mixture.

S.No	AW (%)	Ph (%)	Wt of AW	Wt of Ph
1	100	0	1.3256,0.9532	0
2	90	10	0.9046	0.1010
3	80	20	0.8044	0.2015
4	70	30	0.7010	0.3010
5	60	40	0.6125	0.4030
6	50	50	0.5250	0.5250

5.5 Crystallization of A/W – Ph frit mixture

The 100% AW glass pallet was fired at 900°C in tube furnace for 1hr. the rate of heating was 3°C/min. All other samples were fired at 950°C in tube furnace for 1hr with rate of heating 3°C/min. A/W powder wad also fired at 950°C.

Xrd Analysis was done for all the samples and the desired phase was obtained which is discussed in detail in result and discussion section.

5.6 Determination of apparent porosity and bulk density

Procedure

Dry weight of all the samples were taken and kept in distilled water for 3hrs in vacuum desiccator. Then suspended weight and soaked weight were taken. The required apparent porosity and bulk density were calculated according to following formula:

$$\text{A.P} = \frac{(W-D)}{(W-S)} * 100$$

$$\text{B.D} = \frac{D}{(W-S)}$$

Where,

W = Soaked Wt

D = Dry Weight

S = Suspended Wt

5.7 *Determination of Hardness*

Vickers hardness: It is a measure of the hardness of a material which is calculated from the size of an indentation produced under load by a pyramid-shaped diamond indenter.

Procedure: The diamond is pressed into the surface of the material at of 1kgf with dwell time of 5sec and the size of the impression was measured using calibrated microscope. The machine used was carver press. The Vickers number (HV) is calculated using the following formula:

$$HV = 1.854(F/D^2),$$

Where,

F = Applied Load (measured in kilograms-force) and

D^2 = Area of the indentation (measured in square millimetres).

5.8 Determination of Machinability

As mica crystals possess excellent cleavage planes perpendicular to the c-axis, introduction of phlogophite phase gives machinability to the A/W & Ph mixture and A/W provides the required bioactivity. To check the machinability of the sample, 60:40 & 50:50 glass ceramic were drilled using iron needle at 1700rpm and the samples were drilled up to 80% extent.

CHAPTER 6

Result and discussion

6.1 Determination of Apparent Porosity & bulk Density

Table 4. Calculation of A.P and B.D.

S.No	Ratio of AW & Ph	Apparent Porosity	Bulk Density
1	100:0	0.5069	2.7595
2	90:10	0.5410	2.6773
3	80:20	0.5619	2.4603
4	70:30	0.6226	2.3936
5	60:40	0.9392	2.0973
6	50:50	0.9559	2.0961

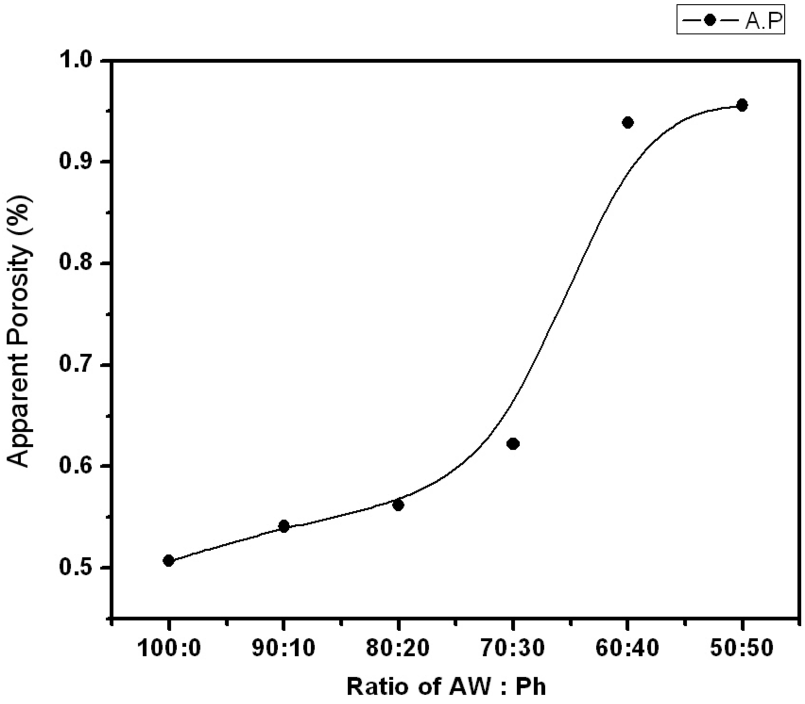

Fig 6. Variation of apparent Porosity with different ratio of AW & Ph.

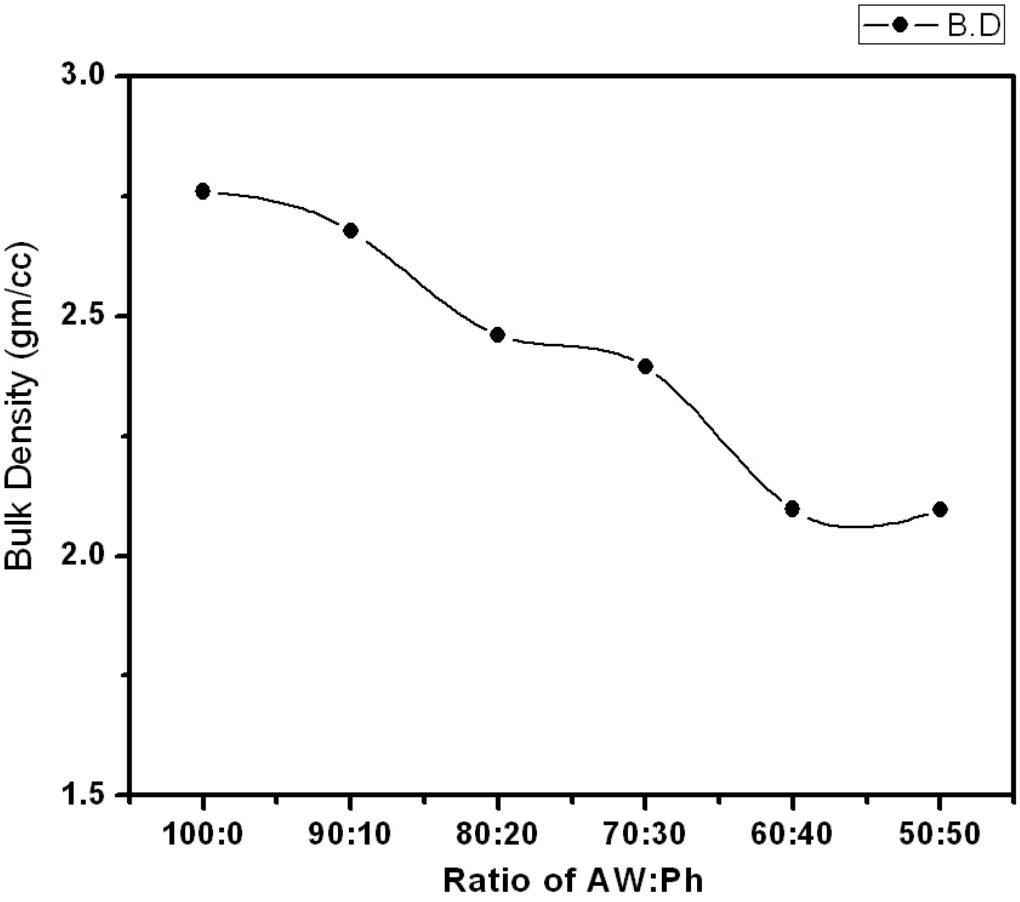

Fig 7. Variation of Bulk Density with different ratio of AW & Ph.

The above A.P & B.D variations shows that apparent porosity increases with the increase in Ph content in the AW matrix on the other hand the bulk density decreases.

6.2 Determination of Hardness:

Table 5. Calculation of Hardness

S.No	**Ratio of AW & Ph**	**D1**	**D2**	**Hardness (Vicker Scale)**	**Avg Hardness**	**Avg Hardness(GPa)**
1	100:0	64.5 63.5	60.5 62.5	474.7 467.2	470.95	4.169
2	90:10	59.8 59.2	58.0 53.3	534.2 532.7	533.45	5.232
3	80:20	63.8 63.3	63.4 63.8	458.4 459.2	458.8	4.499
4	70:30	65.6 64.0	66.9 64.5	422.5 448.5	440.85	4.323
5	60:40	65.2 65.2	65.7 65.8	432.9 432.2	432.55	4.242
6	50:50	65.6 65.1	65.6 65.2	430.9 436.9	433.9	4.255

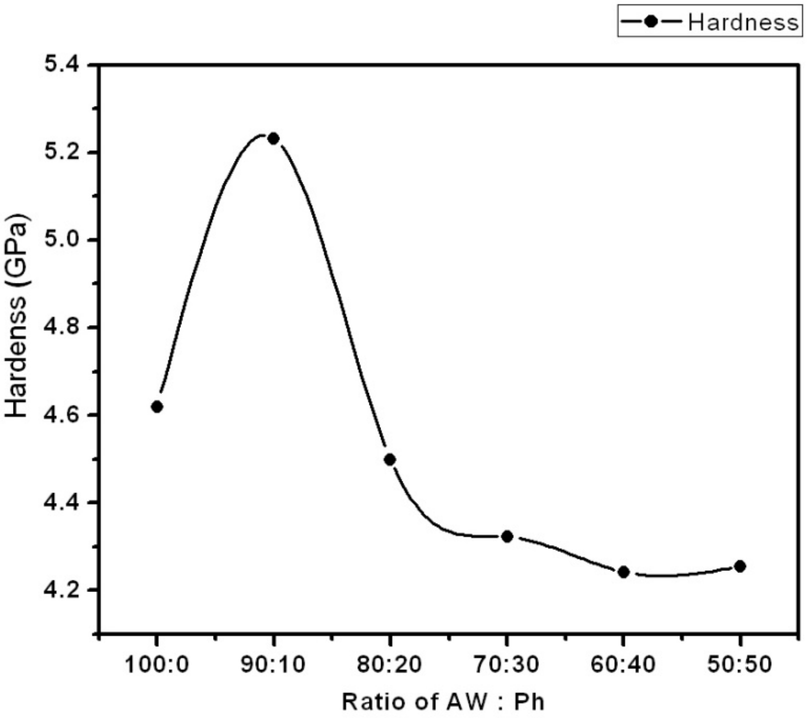

Fig 8. Variation of Hardness with different ratio of AW & Ph.

The hardness decreases with increase in Ph percentage in the matrix of AW except at addition of 10% ph were hardness increases.

6.3Machinability

Two sample of composition 60:40 and 50:60 (AW:Ph) were drilled using driller having rpm of 1700. Both of the samples responded positively to the test initially but the sapmle breaks after drilling upto 80% extent which may be due to uneven phase distribution of the phlogophite phase.

Drilling of samples

a. AW : Ph – 50:50

ew

Fig 9. Drilling of 50:50 Sample.

b. AW : Ph – 60:40

v

le.

6.4X – Ray diffraction pattern

a. 100% AW Fired at 900°C

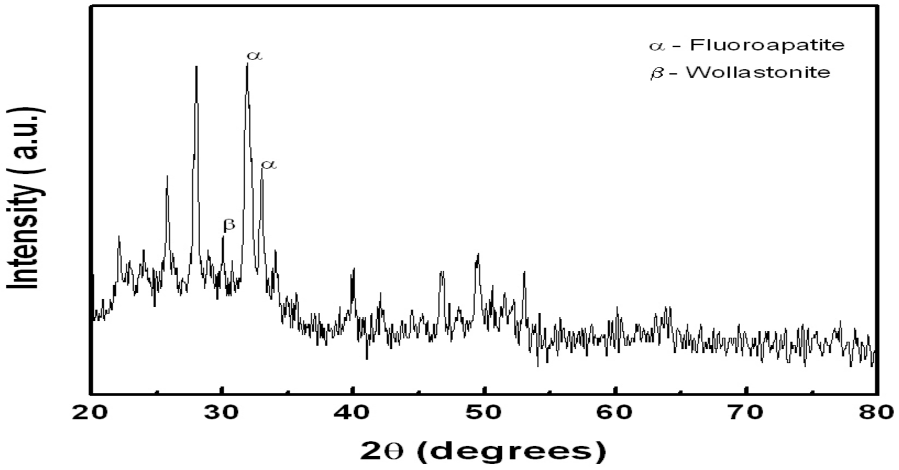

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Fig11. Xrd pattern of 100% AW fired at 900^{o}C.

The Fig11 shows the XRD pattern of 100% AW fired at 900^{o}C. It contains fluroapatite(α) phase, JCPDS - 12-0261 and wollastonite (β) phase, JCPDS - 42-0547.

b. 100% AW Fired at 950°C

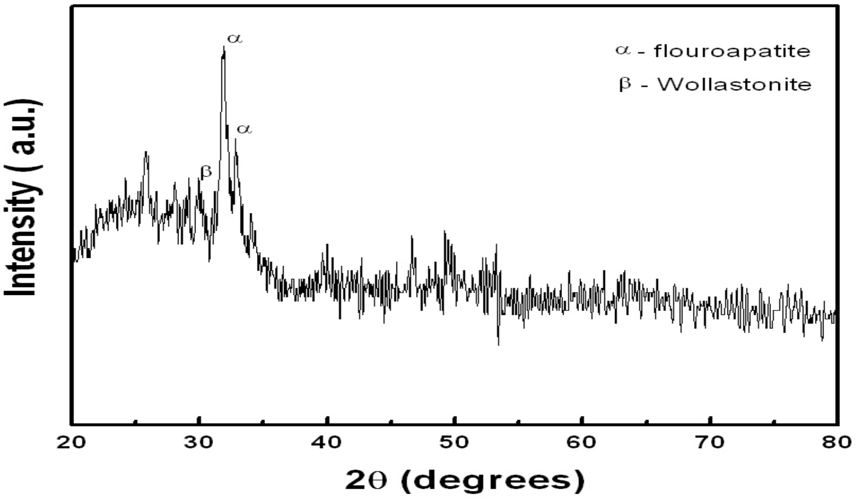

Fig 12. Xrd pattern of 100% AW fired at 950^{o}C.

The Fig shows the XRD pattern of 100% AW fired at 950^{o}C. It contains fluroapatite(α) phase, JCPDS - 12-0261 and wollastonite (β) phase, JCPDS - 42-0547.

c. AW : Ph - 90 : 10 Fired at 950°C

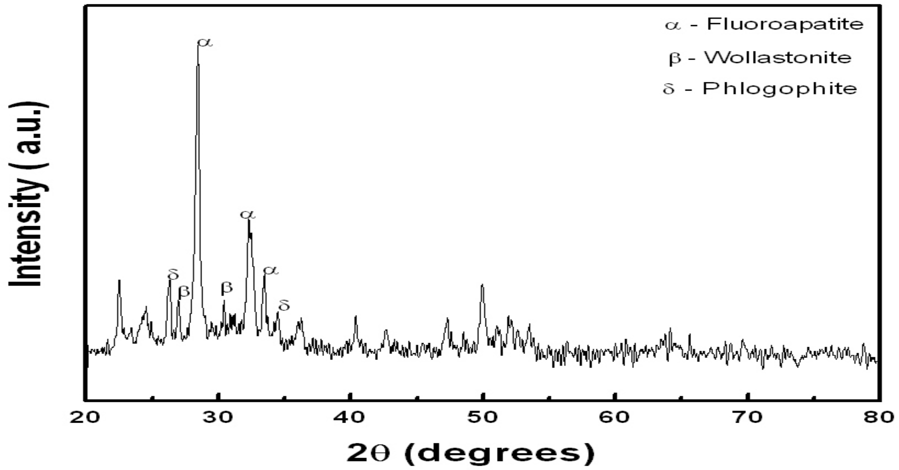

Fig 13. Xrd pattern of AW : Ph – 90:10 fired at 950°C.

The Fig13 shows the XRD pattern of AW : Ph – 90 : 10 fired at 950°C. It contains fluroapatite(α) phase, JCPDS - 12-0261, wollastonite (β) phase, JCPDS - 42-0547 and phlogophite (δ), JCPDS - 76-0816.

d. AW : Ph – 80 : 10 Fired at 950°C

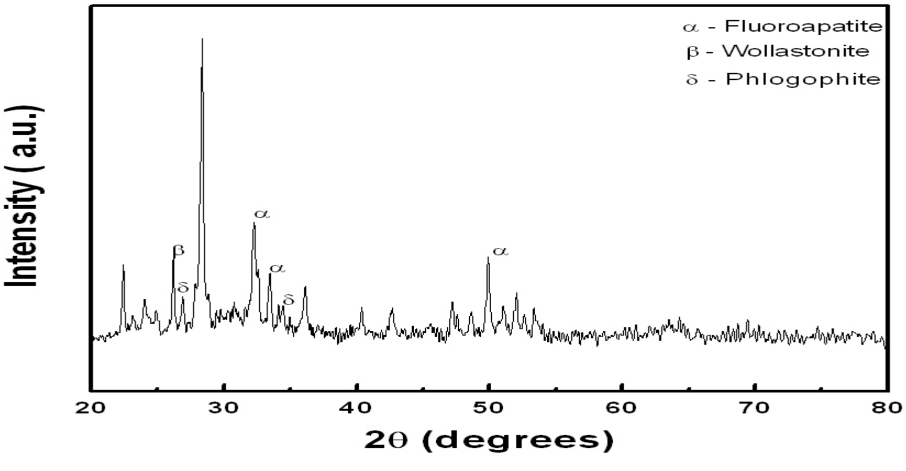

Fig 14. Xrd pattern of AW : Ph – 80:20 fired at 950°C.

The Fig 14 shows the XRD pattern of AW : Ph – 80 : 20 fired at 950°C. It contains fluroapatite(α) phase, JCPDS - 02-0845, wollastonite (β) phase, JCPDS - 72- 2284 and phlogophite (δ), JCPDS - 76-0816.

e. *AW : Ph – 70 : 30 Fired at 950°C*

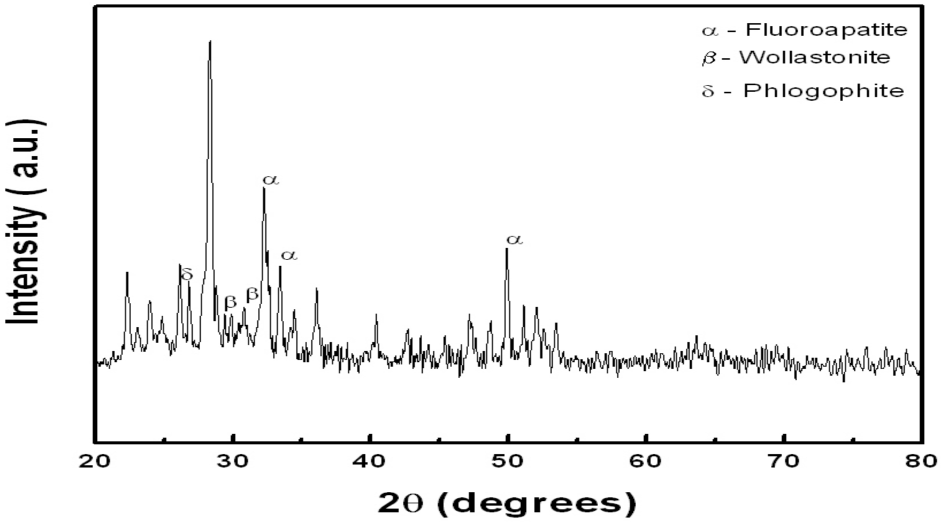

Fig 15. Xrd pattern of AW : Ph – 70:30 fired at 950^{o}C.

The Fig 15 shows the XRD pattern of AW : Ph – 70 : 30 fired at 950^{o}C. It contains fluroapatite(α) phase, JCPDS 20-0845, wollastonite (β) phase, JCPDS - 72- 2284 and phlogophite (δ), JCPDS - 16-0344.

f. AW : Ph – 60 : 40 *Fired at 950°C*

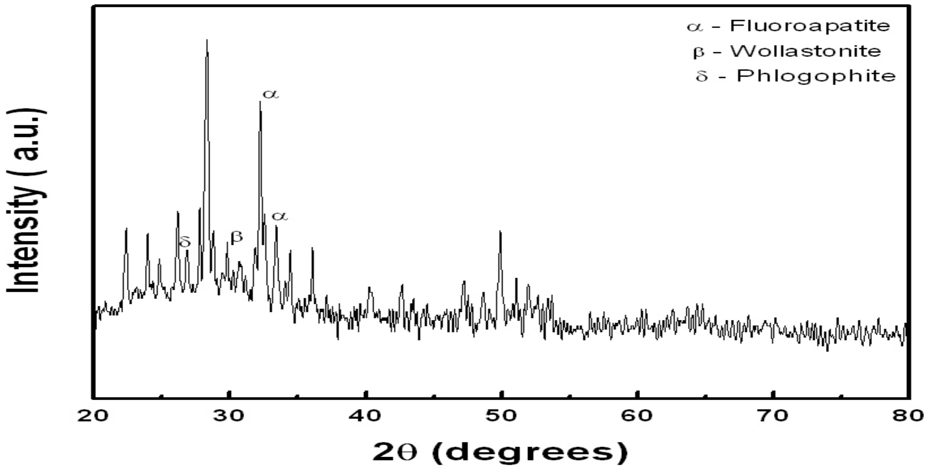

Fig 16. Xrd pattern of AW : Ph – 60:40 fired at 950^{o}C.

The Fig 16 shows the XRD pattern of AW : Ph – 60 : 40 fired at 950^{o}C. It contains fluroapatite(α) phase, JCPDS 71-0880, wollastonite (β) phase, JCPDS - 73-1110 and phlogophite (δ), JCPDS - 16-0352.

g. AW : Ph – 50 : 50 Fired at 950°C

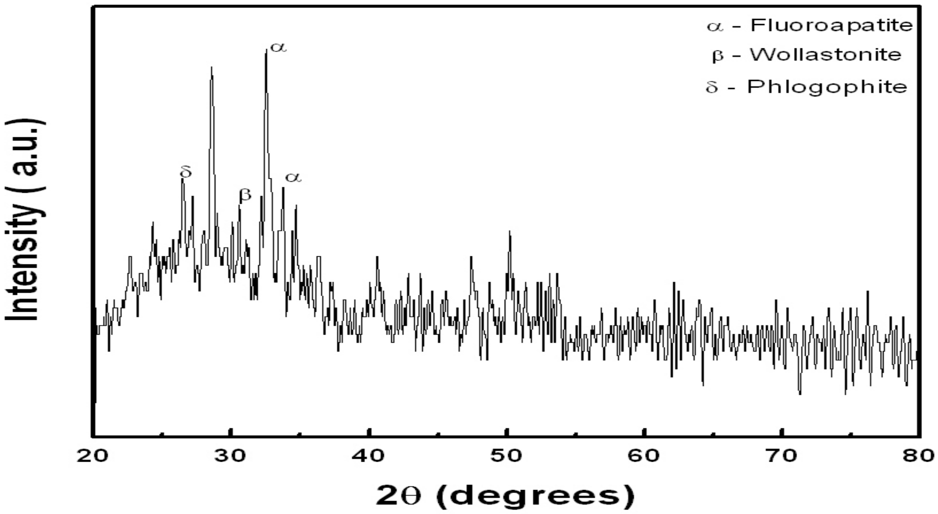

Fig 17. Xrd pattern of AW : Ph – 50:50 fired at 950^{o}C.

The Fig17 shows the XRD pattern of AW : Ph – 60 : 40 fired at 950^{o}C. It contains fluroapatite(α) phase, JCPDS 71-0880, wollastonite (β) phase, JCPDS - 72-2284and phlogophite (δ), JCPDS - 16-0352.

CHAPTER 7

CONCLUSIONS

The incorporation of phlogophite in apatite-wollatonite glass ceramic is mainly intended to increase machineability of AW bioglass to broaden its area of application. The mail conclusion drawn from the present research work may be summarized as under:

1. The apparent porosity increases as the proportion of phlogophite phase increases.
2. Bulk density decreases as the proportion of phlogophite increases as the phlogophite phase has a less density as compared to AW phase.
3. The hardness of the samples decreases with increase in amount of phlogophite.
4. With the incorporation of Ph phase AW glass can be made machinable as demonstrated by the drilling of 60:40 & 50:50 samples. Both the samples break during the last stage of drilling which may be due to uneven phase distribution of the phlogophite phase.
5. As both hardness and bulk density decreases with increase in amount of Ph gives an indication that the machinable properties are being increasing.
6. Optimal proportion of AW & Ph should be taken to achieve both bioactivity and machinability.

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