Effect of thermal treatment on phase and microstructural evolution of magnesium borate ($Mg_2B_2O_5$) nanorod whiskers prepared via hydrothermal cum reaction sintering process

Under the Supervision of-

Dr. Manas Ranjan Majhi (Supervisor)

Ceramic Engineering Department

Presented By-

Vaibhav Singh

Research Scholar

Ceramic Engineering department

IIT (BHU) VARANASI

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Introduction

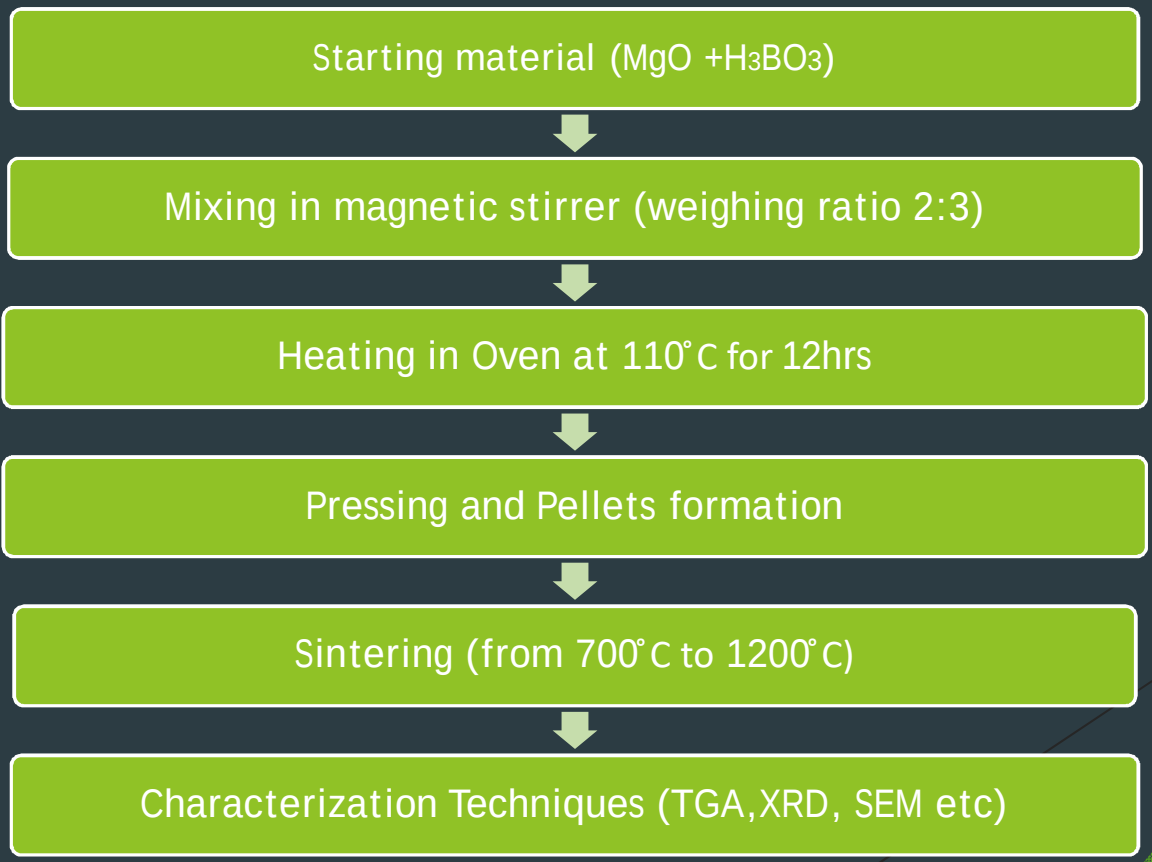
- Whiskers are one dimensional, long, nano-structure materials which are in the form of nanowires, nanorods, nanobelts and nanotubes.
- Now a day's ceramic whisker evolved as a potential reinforcement in the fabrication of metal, ceramic and polymer-based composite because of their wide range of fitting properties.
- Ceramic whisker in particular magnesium borate not only improves mechanical and microstructural properties but also improves thermal and antiwear properties of composites.
- $Mg_2B_2O_5$ whisker has very attractive features as a reinforcement in composites.

Applications

- It is used as a thermo-luminescent phosphor.
- Luminescent material for cathode ray tube screens.
- Catalyst reinforcing material for plastics.
- Excellent resistance for corrosion-erosion in basic oxygen steel making.
- Good thermal shock resistant in fused cast refractories.
- Used as a reinforcement in composites.

Experimental Proceeding

Synthesis Of Magnesium Borate ($Mg_2B_2O_5$) Whisker via hydrothermal process from MgO and H_3BO_3



Synthesis of $Mg_2B_2O_5$ Whisker via Hydrothermal route

STARTING MATERIAL

- The primary raw material used for the synthesis of Magnesium Borate ($Mg_2B_2O_5$) whisker is Mg0 and Boric Acid.

SEIVING

- This is done by passing the raw material from sieve shaker of 45 micron size separately.

Direct Mixing in magnetic stirrer

- First of all we have to take 50 gm sample of MgO and Boric acid in a beaker. Add 300 ml of distilled water in it.
- Then the beaker is placed on a magnetic stirrer for stirring the solution for 2 hr at 80℃.
- After stirring at 80℃, the solution is dried into oven at 110℃ for 12 hrs.

BINDER ADDITION

- The 5% w/w PVA binder was slowly added after mixing in auger mortar pestle.

PRESSING AND PELLET FORMATION

- Pressing is done on the hydraulic pressing machine for pellet formation.
- In this the powder mixtures were uniaxially compacted into rectangular and circular pellet specimen in a high carbon chrome die.
- Which were further compacted by cold isostatic pressing at 10 Tons on hydraulic pressing machine.

SINTERING

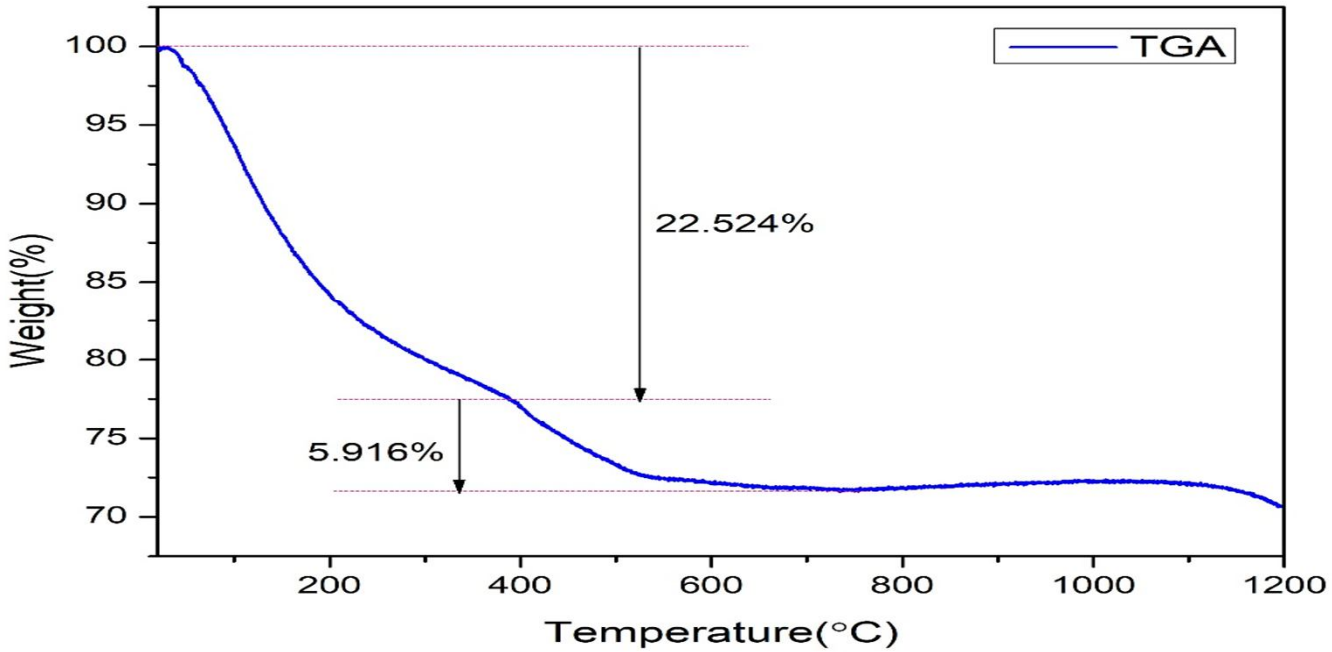
The pellets were sintered in an oxide electric furnace from 700 to 1200°C sintering temperature, with 5°C/min heating rate and soaking for 4 hours.

Pellet after sintering at 1200°

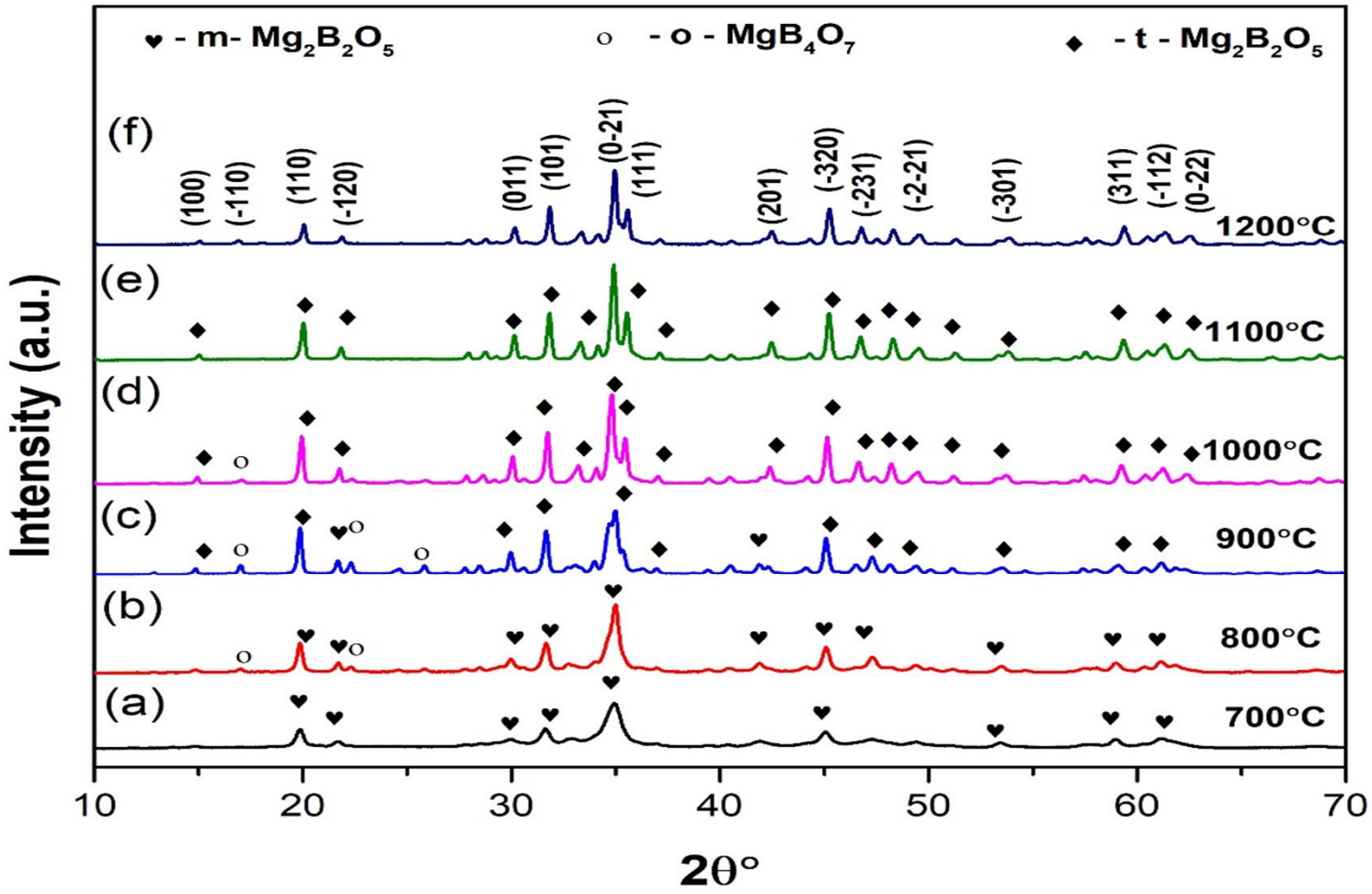
Oxide Electric Furnace

Results and Discussion

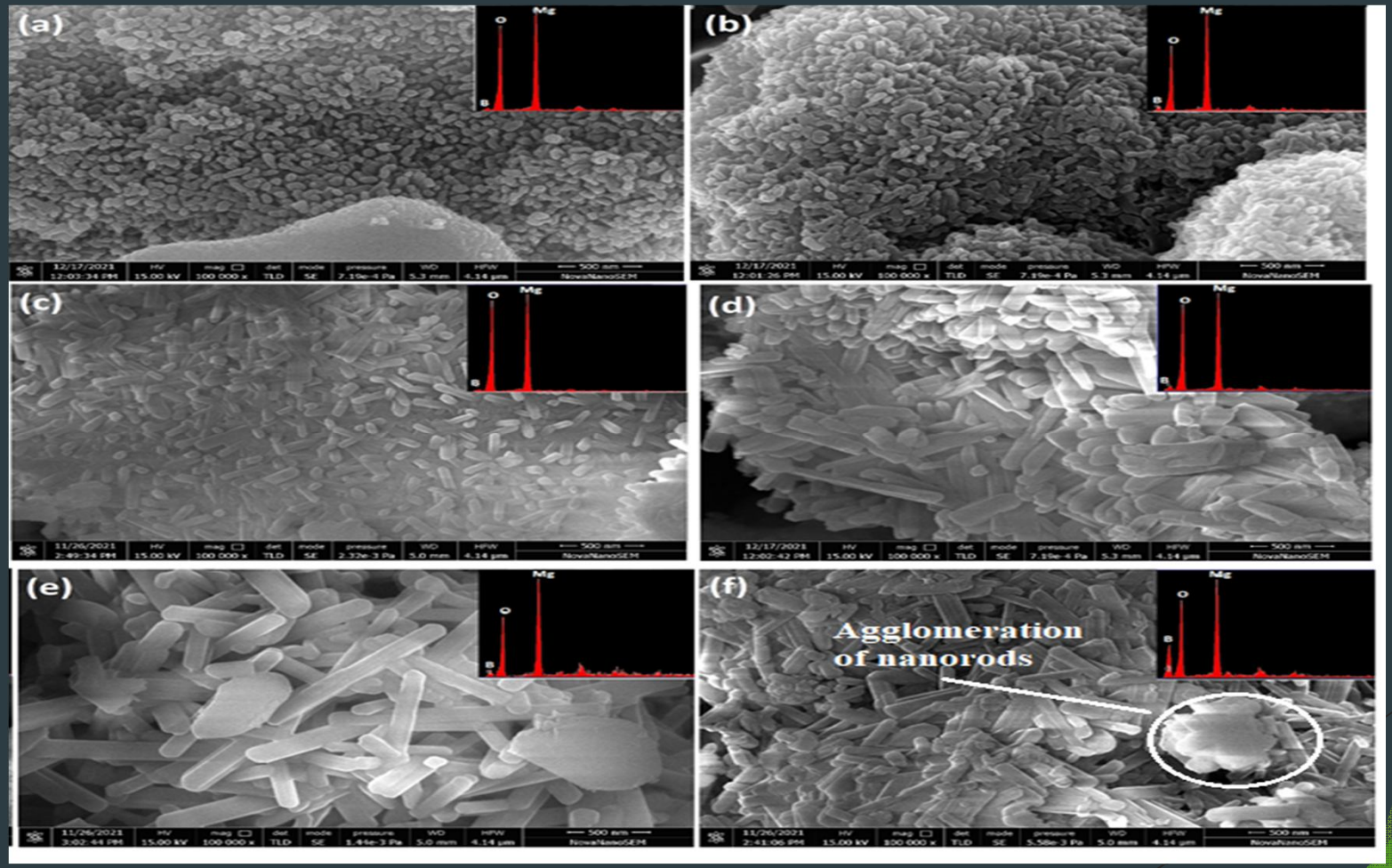
TGA Analysis



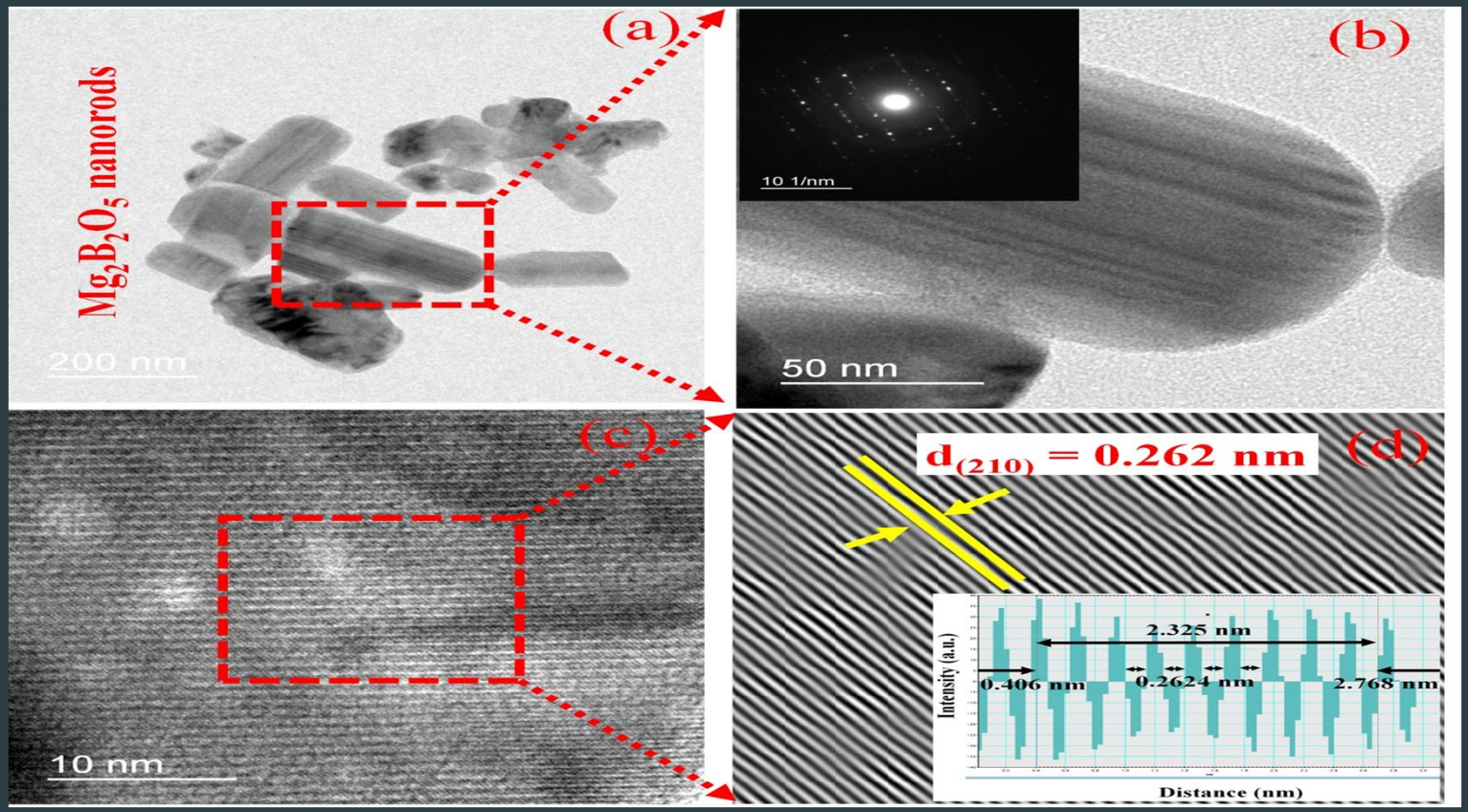
XRD analysis of synthesized magnesium borate



HRSEM of synthesized magnesium borate



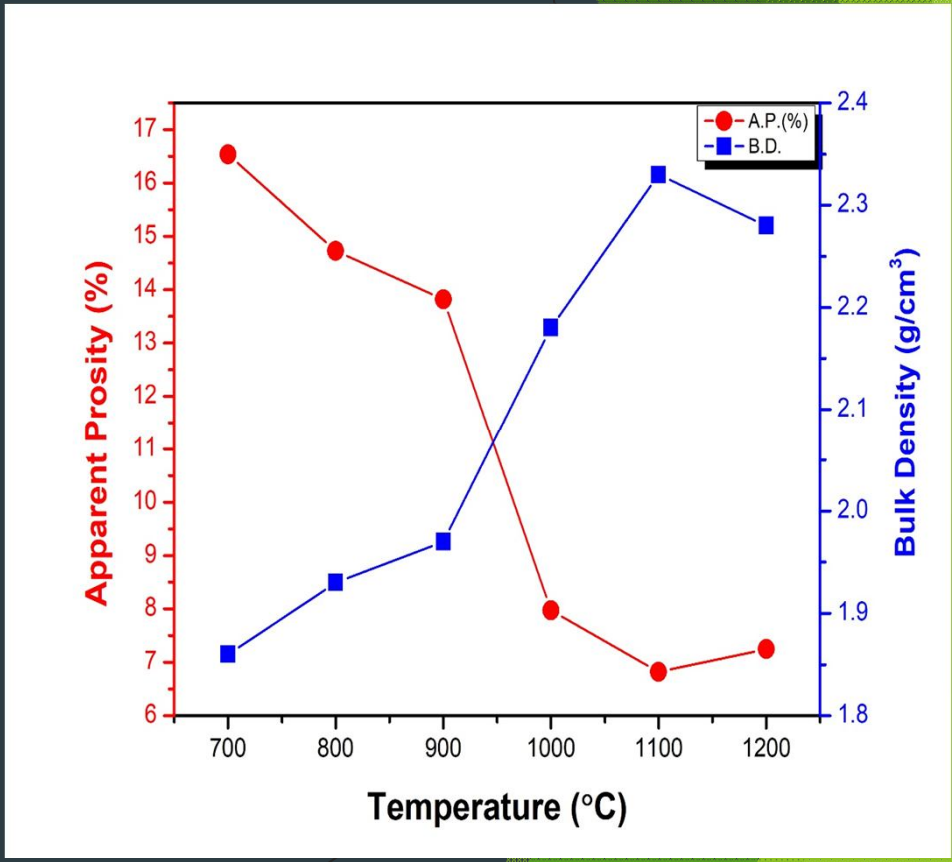
TEM



(a) TEM image shows $Mg_2B_2O_5$ nanorod structure; **(b)** magnified TEM and corresponding SAED pattern (inset) of $Mg_2B_2O_5$ nanorod at 1200 °C; **(c)** HRTEM; **(d)** calculation of d-spacing using IFFT (inset) in Gatan software

Physical properties of sintered sample

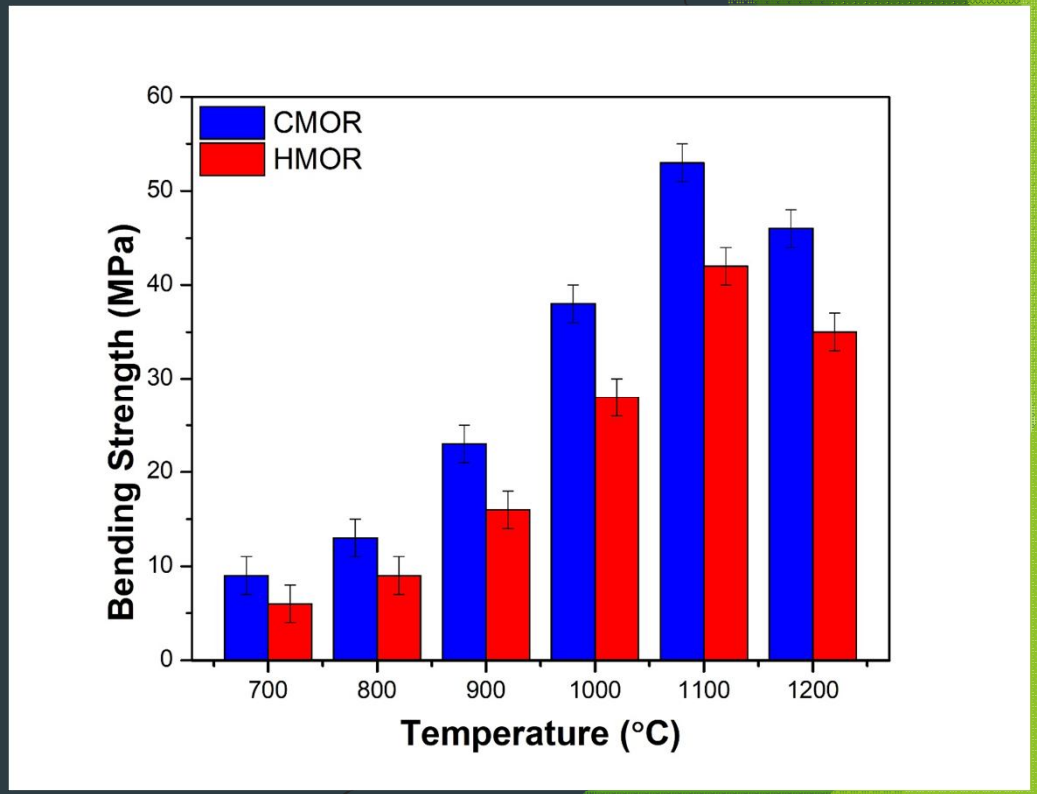
Processing temperature (°C)	Apparent Porosity (%)	Bulk density (g cc^{-1})
700	16.54	1.86
800	14.73	1.93
900	13.82	1.97
1000	7.97	2.18
1100	6.82	2.33
1200	7.25	2.28



Mechanical Testing

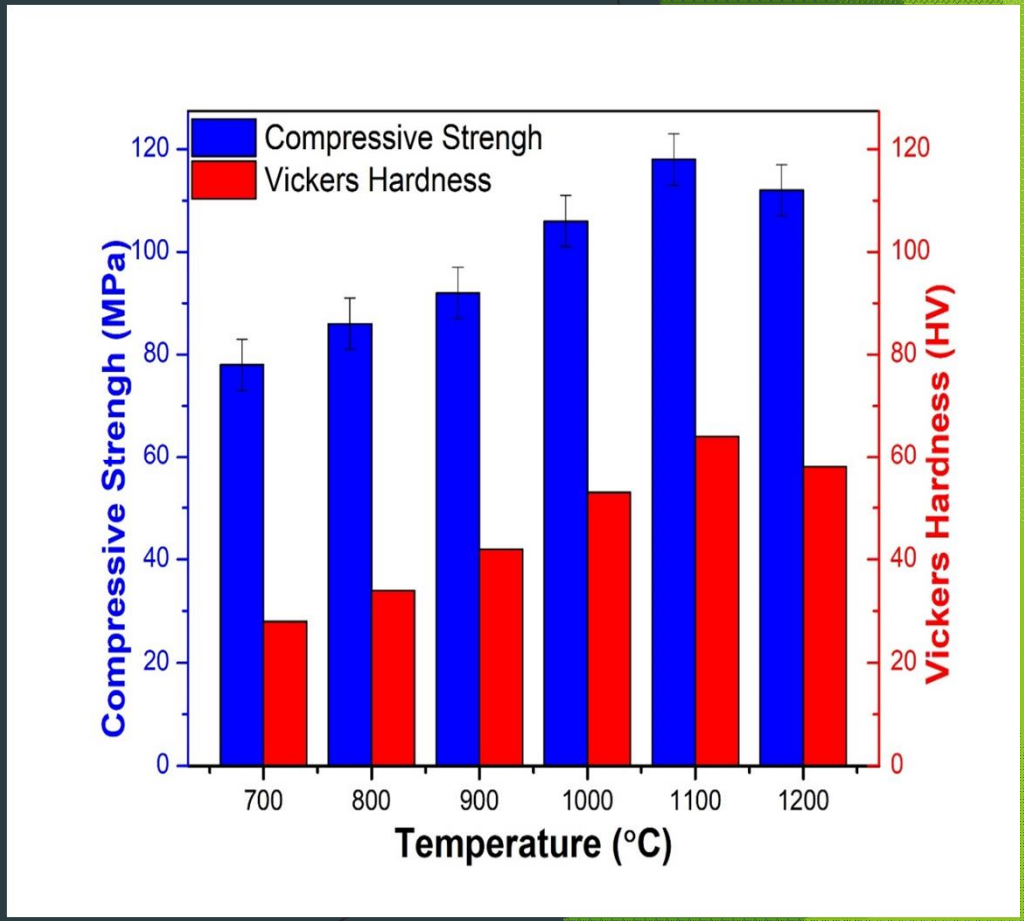
Bending strength at room temperature (CMOR) and at 800°C (HMOR)

Processing temperature (°C)	Bending strength at ambient condition (CMOR) (MPa)	Bending strength at 800 °C (HMOR) (MPa)
700	9 ± 2	6 ± 2
800	13 ± 2	9 ± 2
900	23 ± 2	16 ± 2
1000	38 ± 2	28 ± 2
1100	53 ± 2	42 ± 2
1200	46 ± 2	35 ± 2



Compression and Hardness Test

Processing temperature (°C)	Compressive Strength (MPa)	Vickers Hardness (HV)
700	78 ± 5	28
800	86 ± 5	34
900	92 ± 5	42
1000	106 ± 5	53
1100	118 ± 5	64
1200	112 ± 5	58



Conclusion

- High purity single crystalline $Mg_2B_2O_5$ nanorod whiskers were successfully synthesized via hydrothermal cum reaction sintering process.
- The structure of $Mg_2B_2O_5$ changes from monoclinic to orthorhombic followed by triclinic when the sintering temperature increases from 700 to 1200°C, as analyzed by XRD results.
- SEM and TEM images confirm the formation of circular cross-section nanorod like ceramic whisker having average diameter varies between 47 to 130 nm and average length varies between 63 to 715 nm at sintering temperature ranging from 700 to 1200°C.
- The SAED pattern reveals that these nanorods are single crystals and HRTEM indicates the formation of triclinic phase of $Mg_2B_2O_5$ nanorods at 1200°C sintering temperature.
- Physical and mechanical properties of synthesized $Mg_2B_2O_5$ was investigated between 700 to 1200°C sintering temperature. It was found that sintering temperature of 1100°C as an optimum temperature for the synthesis of nanorod whiskers.

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THANK YOU