



Eskişehir Technical University
Materials Science and Engineering Department

MLZ 331

Materials Processing
Laboratory-I

2023 – 2024 FALL

Course Instructors

Prof. Dr. Ferhat KARA
Prof. Dr. Semra KURAMA
Prof. Dr. Alpagut KARA
Assoc. Prof. Dr. Ali ÇELİK
Assist. Prof. Dr. İrfan TÖRE
Assist. Prof. Dr. Emrah DÖLEKÇEKİÇ

Laboratory Instructors

Res. Assist. Dr. Salih Çağrı ÖZER
Res. Assist. Dr. Kübra GÜRCAN BAYRAK
Res. Assist. Burak DEMİR
Res. Assist. İlhan KAHRAMAN
Res. Assist. Özlem Başak ÖZKAN KOLÇUBAŞI
Res. Assist. Levent KÖROĞLU
Res. Assist. Enes İbrahim DÜDEN
Res. Assist. Ertuğrul İŞLEK
Res. Assist. Emine ERSEZER

Course Coordinators

Res. Assist. Dr. Kübra GÜRCAN BAYRAK

GENERAL INSTRUCTIONS FOR MLZ 331 MATERIALS PROCESSING LABORATORY-I

1. Because the experiments are not synchronized with the classroom lectures (i.e., Ceramic Processing), **it is extremely important that you read each experiment and basic references prior to the lab.** The lab instructor will ask general questions during the lab to test your understanding of the lab.
2. The data, calculations, observations, results and graphs are to be recorded directly, into the lab. report.
3. **Before each lab.,** students should **watch the video of corresponding experiment.** The link of video list including all videos separately:

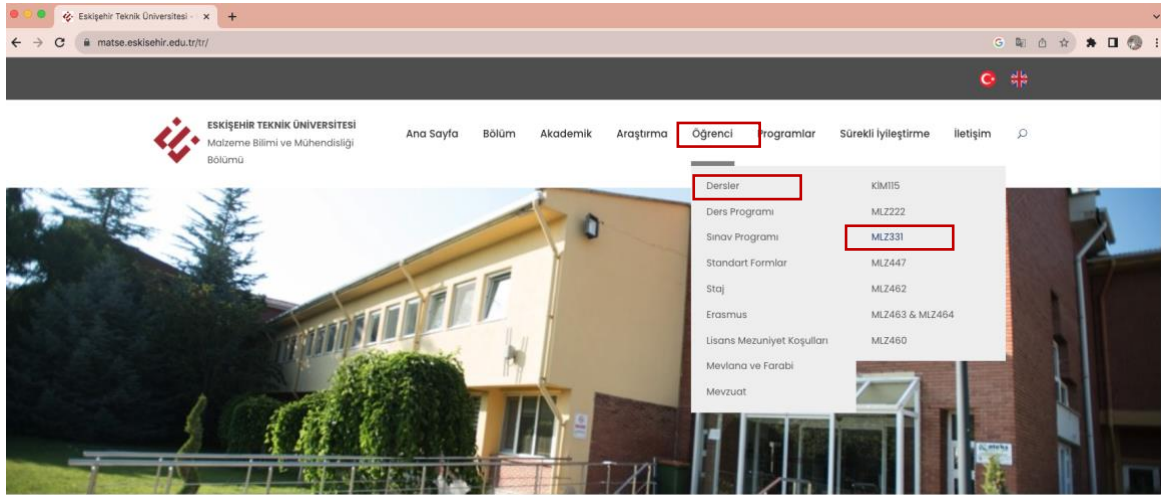
<https://www.youtube.com/playlist?list=PLQUb7RtOnw8KWX-URfjlgjdZ7qeKB2ek>

4. **At the beginning of experiments,** there will be **a quiz composed of 2-3 questions.**
5. It is necessary to prepare a report from the results obtained at the end of the experiments. These reports will be a digital copy (MS Word or PDF document), and it will be sent to www.turnitin.com. Upload your files (firstly, you need to be logged in) with **40924063 class ID and Mlz331 enrolment key.**
 - Files should be uploaded by the name format of **GroupName_Student Number_Name&Surname** (For exp.; **GroupA_12345678910_AhmetYilmaz**).
6. Students are responsible to submit their reports to Turnitin class **on the same day until 17.00 a week after the experiment.** The lab instructor will grade the reports within a week and will post the results. If you wish to discuss the grade, make an appointment to see the lab instructor at his convenience. A copy of the graded reports will be handed to you upon your request if needed.

MLZ331 Materials Processing Laboratory I					+ Add Assignment
	START	END	POST	STATUS	ACTIONS
Experiment 1					
PAPER	09-Oct-2023	29-Oct-2023	N/A	0/0 0%	View More actions
Experiment 2a&b					
PAPER	23-Oct-2023	03-Nov-2023	N/A	0/0 0%	View More actions
Experiment 5a&b					
PAPER	27-Nov-2023	08-Dec-2023	N/A	0/0 0%	View More actions
Experiment 3					
PAPER	30-Oct-2023	19-Nov-2023	N/A	0/0 0%	View More actions
Experiment 4					
PAPER	06-Nov-2023	27-Nov-2023	N/A	0/0 0%	View More actions
Experiment 6					
PAPER	04-Dec-2023	15-Dec-2023	N/A	0/0 0%	View More actions
Experiment 7					
PAPER	11-Dec-2023	22-Dec-2023	N/A	0/0 0%	View More actions

7. The nature of working in groups implies that there should be cooperation and discussion between members of the group and the lab instructor. It is, however, expected that when students prepare their reports, that they do so individually using their own words and interpretation. Plagiarizing or blatant copying of a report or reference will result in an automatic zero for that lab for the first offense. A second offense will result **in an automatic FF grade for the course.**

8. Students must attend each lab on the specified date unless arranged differently with the course instructor. Reports must be handed in on time; **otherwise 10% will be deducted from the mark for each day late.** If there are extenuating circumstances for a report not being completed on time or for not attending a lab, the student should phone or make an appointment with one of the course instructors.
9. For the face to face lessons, it is obligatory to **wear laboratory apron, safety glass, gloves and dust mask unless the lab instructor tells otherwise.** Students without aprons (lab coats) and masks will not be admitted to the laboratory.
10. The lab groups must be **present in the room/building** where the lab will take place (stated in the lab manual) **5 minutes before the lab** starts. Students are obliged to **learn the location of the labs** before the labs begin.
11. Lab manuals will be available **on the department web-sites.**
<https://matse.eskisehir.edu.tr/tr/Icerik/Detay/mlz331-2>



12. Instructions for INFORMAL Lab Reports

- a) For the informal report, it will be prepared according to the lab instructor's content. The requests of the lab instructors may be different from each other. However, all reports should include the following items:

1. Student Name-Surname
2. Student Number
3. Experiment Number
4. Group
5. Date

- b) The reports must be written ***in English*** and will be prepared by the use of computer.
- c) The reports will not exceed **"2 PAGES"**, excluding Tables and Figures. Reports exceeding 2 pages will not be taken into account **(unless the instructor requests otherwise).**
- d) The reports will cover only the **activities performed during the laboratory hour, comments, and the answers to the lab instructor's questions and demands, if any.**

13. Instructions for FORMAL Lab Reports

The formal report is for **Experiment #4 “Dry Pressing”**. The style of the report is based on the type of “Engineering Report”, a professional engineer would have to complete in a working environment. The Lab instructors will share the details of the report with you but, it should be typed and consist of:

- i. **A Title Page**
- ii. An **Abstract** that clearly summarizes the aims of experiment
- iii. **A Table of Contents**
- iv. An **Introduction** section that briefly introduces the concept and its important parameters and clearly states the aims of the experiment.
- v. **A Background** section that briefly discusses experimental procedure and discuss important parameters.
- vi. An **Experimental Procedure** section that carefully summarizes the methods used, the material(s) studied and the apparatus used. Any problems related with compaction stages?
- vii. **A Results** section. Give your results by comparing them with literature.
- viii. **A Discussion of Result** section. Discuss what you expected and what you obtained.
- ix. **Conclusions**-A brief synopsis of your results.
- x. **References** (You can reach the publications via Google Scholar.)
- xi. **Appendix**-raw data, calculations, etc., i.e., items that may be of interest, but not always necessary (usually make reading report difficult).

14. The course grade will be based on the following:

GRADING TABLE

Exam	Exam Type	Percentage of Exam
SHORT EXAMS	Exp#1, Exp#2, Exp#3, Exp#4, Exp#5, Exp#6, Exp#7 Quizzes	20 %
PRACTICE	Formal Report Experiment #4 “Dry Pressing”	15%
2. MIDTERM	Exam	25 %
FINAL	Exp#1, Exp#2, Exp#3, Exp#5, Exp#6, Exp#7 REPORTS	40%

SCHEDULE

Date of the Week	Laboratory Instructors	Experiment Name	Lab Locations
05.10.2023	Res. Assist. Dr. Kübra GÜRCAN BAYRAK	Team Meeting	
09.10.2023- 13.10.2023	Res. Assist.Dr. Kübra GÜRCAN BAYRAK Res. Assist. Enes İbrahim DÜDEN	Experiment#1 Raw Material Preparation and Particle Size Analysis	MLZ 123 Ceramic Process Lab I (Seramik Süreçler Lab I)
16.10.2023- 20.10.2023	Res. Assist. Burak DEMİR Res. Assist. Levent KÖROĞLU	Experiment #2a Particle Dispersion and Slip Casting - I	MLZ 123 Ceramic Process Lab I (Seramik Süreçler Lab I)
23.10.2023- 27.10.2023	Res. Assist. Burak DEMİR Res. Assist. Levent KÖROĞLU	Experiment #2b Particle Dispersion and Slip Casting - II	MLZ 123 Ceramic Process Lab I (Seramik Süreçler Lab I)
30.10.2023- 3.11.2023	Res. Assist. Ertuğrul İŞLEK	Experiment #3 Tile Production	MLZ 123 Ceramic Process Lab I (Seramik Süreçler Lab I)
06.11.2023- 10.11.2023	Res. Assist. İlhan KAHRAMAN Res. Assist. Emine ERSEZER	Experiment #4 Dry Pressing	MLZ 123 Ceramic Process Lab I (Seramik Süreçler Lab I)
Midterm Exams (13.11.2023-25.11.2023)			
27.11.2023- 01.12.2023	Res. Assist. Dr. S.Çağrı ÖZER Res. Assist. Ertuğrul İŞLEK	Experiment#5a Sintering of Ceramics Experiment#5b Density and Porosimetry	MLZ 122 Advanced Ceramic Lab (İleri Tek. Seramik. Lab) MLZ 117 X-Rays Lab (X-Işınları Lab)
04.12.2023- 08.12.2023	Res. Assist. Emine ERSEZER	Experiment #6 Glaze Preparation and Application	MLZ 123 Ceramic Process Lab I (Seramik Süreçler Lab I)
11.12.2023- 15.12.2023	Res. Assist. Ö.Başak ÖZKAN KOLCUBAŞI Res. Assist. Enes İbrahim DÜDEN	Experiment #7 Thin Film Production	MLZ 129 Thin Film Lab (İnce Film Lab)
Final Exams (08.01.2024-20.01.2024)			

GROUPS

Monday	09:00 / 11.00	“Group E”	Assoc. Prof. Dr. Ali ÇELİK
Tuesday	16:00 / 18:00	“Group G”	Prof. Dr. Semra KURAMA
Wednesday	11.00 / 13.00	“Group B”	Prof. Dr. Alpogut KARA
Thursday	09.00 / 11.00	“Group A”	Assist. Prof. Dr. İrfan TÖRE
Friday	09.00 / 11.00	“Group D”	Prof. Dr. Ferhat KARA
Friday	13.00 / 15.00	“Group C”	Assist. Prof. Dr. Emrah DÖLEKÇEKİÇ

EXPERIMENT 1 (MLZ331) RAW MATERIAL PREPARATION AND PARTICLE SIZE ANALYSIS

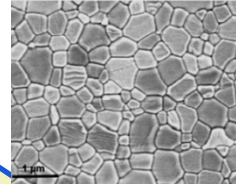
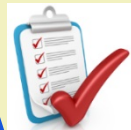
1. Objective of the Experiment

- To learn industrial techniques for size reduction and homogenization of raw materials
- To measure particle size and surface area of ceramic powders with traditional and advance characterization equipments

2. What you should know before the experiment?

You should know;

- 📖 Sampling methods and their usage (Alternative Shovel, Cone and Quartering, Splitting methods, Dividing by spatula)
- 📖 Particle size reduction methods (Crushing, grinding, milling)
- 📖 Milling types, equipments and parameters (wet milling, dry milling, gyratory mill etc, milling jar etc...)
- 📖 Common particle size measurement methods (Sieving, Laser Diffraction, BET, sedimentation etc.)
 - 📖 Laser diffractometer principle
 - 📖 BET principle
 - 📖 Microscopic techniques (SEM, etc)



3. What will you learn during the experiment?

You will learn;

- ✂ How to prepare representative samples?
- ✂ How to carry out dry and wet milling? What are the important parameters?
- ✂ How to determine particle size distribution by sieving and laser diffraction methods?
- ✂ What is the description of the Mesh Size and Equivalent Sphere Approach and their importance?
- ✂ How to measure specific surface area of a powder by using BET equipment?

Preparation of representative sample



Wet milling by ball mill



Dry milling by gyratory mill



Particle Size analysis by dry sieving



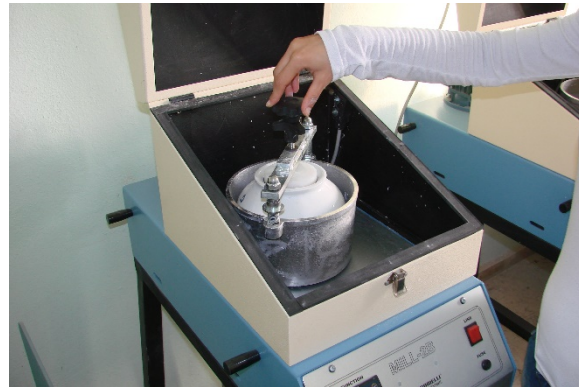
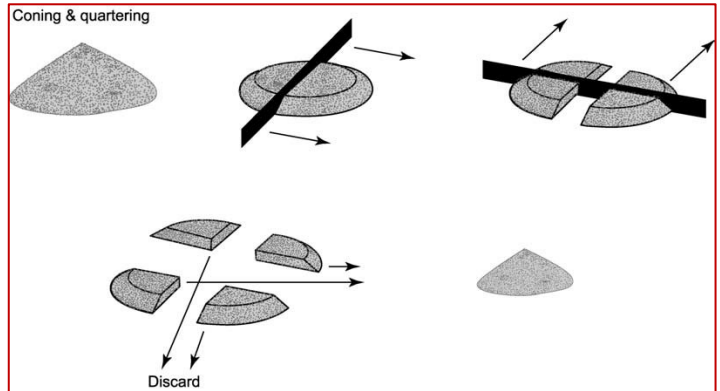
Particle Size analysis by using Laser Diffraction instrument



Demonstration of particle size analysis by BET equipments

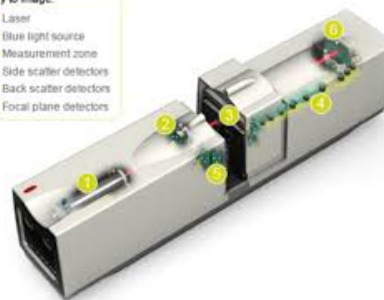


Explanation of particle size analysis by microscobic techniques



Key to image:

- 1 Laser
- 2 Blue light source
- 3 Measurement zone
- 4 Side scatter detectors
- 5 Back scatter detectors
- 6 Focal plane detectors



5. Equipments and materials (if necessary user manuals)

For Raw Material Preparation;

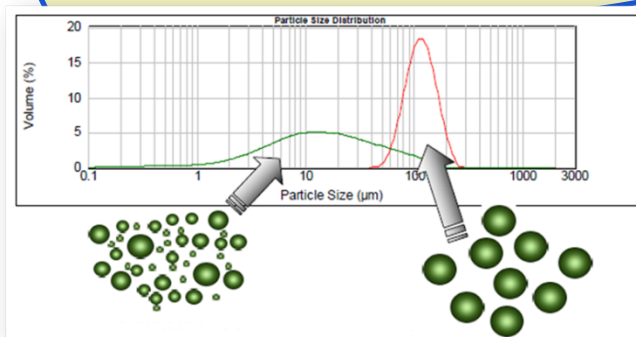
- Raw materials, water, balance, shovel, ruler, splitter, gyratory mill, ball mill.

For Particle Size Analysis;

- Raw materials, balance, water, sieves with different mesh numbers, sieving equipment, laser diffraction instrument, BET equipment.

6. Important points / hints for the equipments and/or results obtained from the analyses

- Why particle size control is important and why particle size analysis should be performed
- Using complementary techniques (e.g, laser diffraction and SEM) for precise characterization of particle size and size distribution
- Choosing appropriate milling and particle size analysis techniques
- Reasonable interpretation of the results



7. Content of the report

- Please draw a flow chart that you learn during the experiment in detail.
- Please explain the particle size result of your sample that you measure by the laser diffraction method.



8. Questions

1. What is the importance of particle size control in material processing?
1. Is it possible to include information about the particle size of a material by measuring the gas adsorption of material?








1. Objective of the Experiment

To achieve basic knowledge on rheology of materials and particle dispersion in ceramics, prepare sanitaryware slips and determine the casting properties depending on the flocculation behavior of slip.

2. What you should know before the experiment?





You should know;

-  Definitions of Newtonian, dilatant, pseudoplastic, thixotropic and Bingham plastic behaviors.
-  What is Brownian Motion?
-  What is zeta potential?
-  What are electrostatic, steric and electrosteric stabilizations?
-  General concepts about ceramic forming techniques.



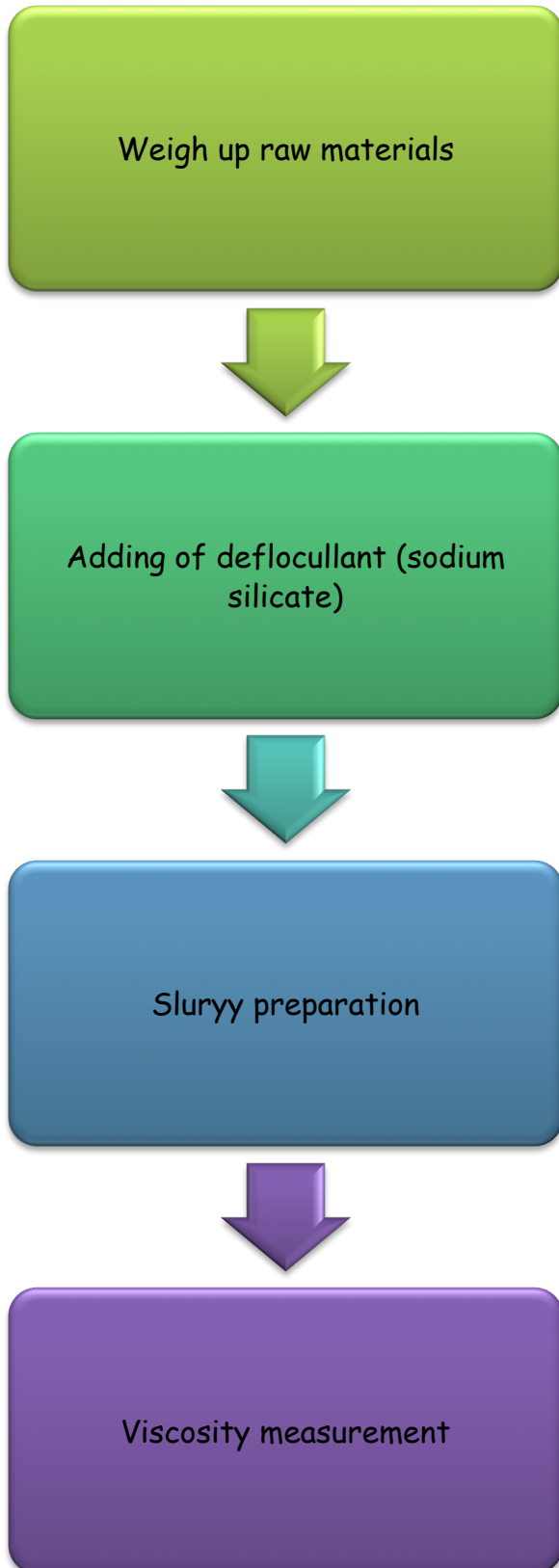
3. What will you learn during the experiment?

You will learn;

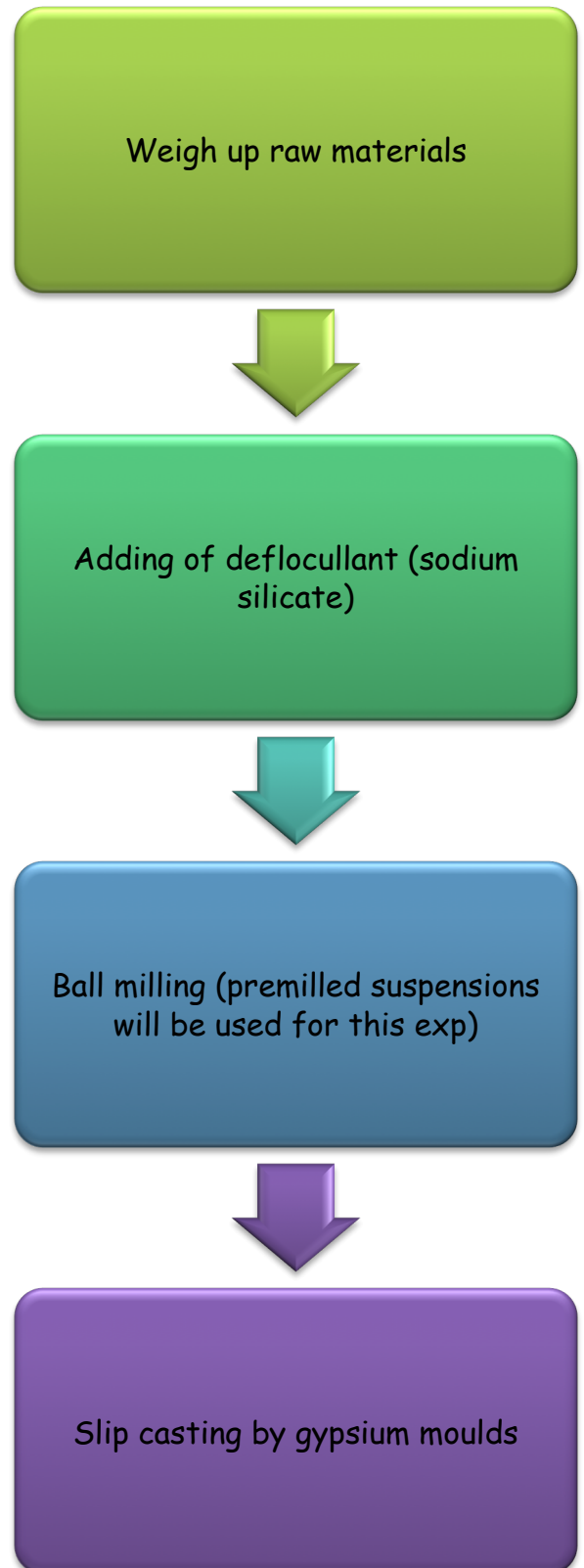
-  How to stabilize ceramic suspensions?
-  How to characterize/estimate the degree of stabilization?
-  How to form ceramic bodies by slip casting?
-  How deflocculation of slurry affects the green body?

4.Schematic view of experimental procedure

Particle dispersion



Slip preparation and Slip casting



The coefficient of **viscosity**, η (Pa.s) indicates the resistance to flow due to internal friction between the molecules of the liquid. A shear rate, $\dot{\gamma}$ (1/s) is required to initiate and maintain laminar flow in a sample liquid. When a shear stress, τ (Pa) is linearly dependent on the velocity gradient (shear rate), liquid shows **Newtonian behavior**.

$$\tau = \eta \cdot \dot{\gamma}$$

where τ is shear stress (Pa); η is viscosity (Pa.s); and $\dot{\gamma}$ is shear rate (1/s).

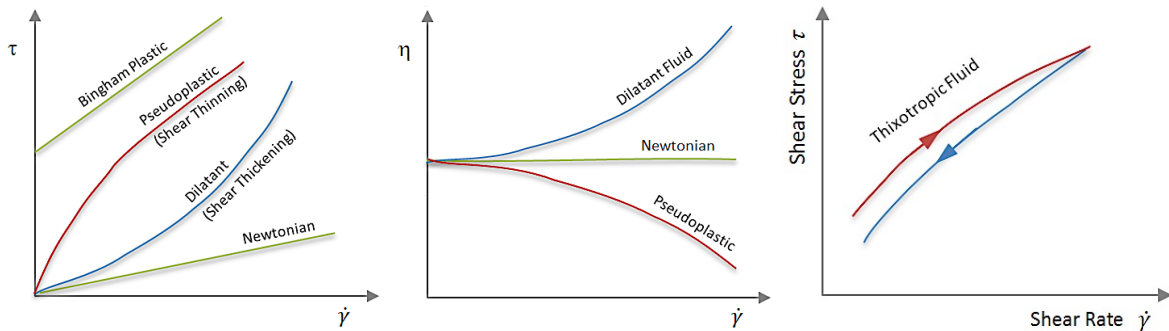
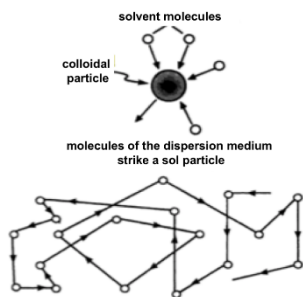


Figure 1. Schematic a) flow and b) viscosity curves of Newtonian and non-Newtonian materials; and c) flow curve of thixotropic fluid

Non-Newtonian materials provide a nonlinear dependence of shear stress on shear rate. If the viscosity decreases with increasing shear rate, behavior is said to be **shear thinning (pseudoplastic)**. In terms of the flow behavior of moderately concentrated suspensions containing large agglomerates and deflocculated slurries, viscosity increases with an increase in the shear rate. This dependence on shear rate is called **shear thickening (dilatant)** behavior.

Slurries containing a linkage of bonded molecules and particles require a finite stress called the yield stress, τ_Y to initiate flow. This type of behavior is known as **Bingham plastic behavior**. The apparent viscosity of a Bingham plastic is higher when the yield stress is higher, and that decreases with increasing shear rate. Unlike to pseudoplastic behavior, shear rate - shear stress data follow a straight line with a yield stress.

The rheological behavior discussed above was assumed to be independent of the shear history and shearing time. For some materials the apparent shear resistance and viscosity at a particular shear rate may decrease with time. This behavior, called **thixotropy**, is commonly observed for shear-thinning materials when the orientation and bonding (coagulation) of molecules or particles change with the time during shear flow.



Colloidal particles in a sol are continuously bombarded by the molecules of the dispersion medium on all sides. The impacts are however not equal in every direction. As a result, the sol particles show random or zig-zag movements. This random or zig-zag motion of the colloidal particles in a sol is called **Brownian motion**.

Figure 2. Brownian motion of colloidal particles

The phenomenon of Brownian motion was observed by Robert Brown in the form of random zig-zag motion of pollen grains suspended in water. This kind of movement is found in all colloidal systems. The Brownian motion becomes progressively less prominent, as the particles grow in size or the viscosity of the medium increases.

Charged particles in a suspension will respond to an imposed potential difference. During flow, a slippage plane must occur somewhere in the electrical double layer. The potential at the slippage plane is called the **zeta potential (ζ -potential)**.

Electrostatic Stabilization

In the electrostatic stabilization of aerosols, the Coulombic repulsion between the colloidal particles is of a long range character and can impart stability. In liquid dispersion media, however, the principle of electroneutrality demands that the net charge in the dispersion medium be equal, but opposite in sign, to that of particles. This leads to a more rapid fall-off in the potential. The counterions in the dispersion medium, however, give rise to the electrical double layers that surround the colloidal particles. It is the mutual repulsion of these double layers that provides stability in electrostatic stabilization.

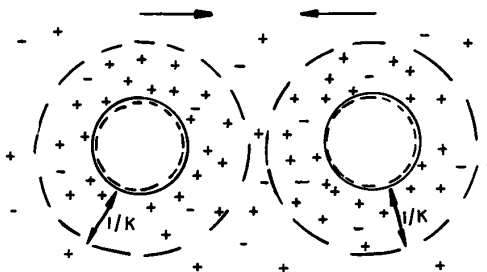


Figure 3. Diagrammatic representation of the origin of electrostatic stabilization for negatively charged particles.

Steric Stabilization

Steric stabilization of colloidal particles is imparted by macromolecules that are attached (e.g. By grafting or by physical adsorption) to the surfaces of the particles.



Figure 4. Schematic representation of steric stabilization.

Electrosteric Stabilization

It is possible to have combinations of electrostatic and steric stabilization, which has been termed electrosteric stabilization. The electrostatic component may originate from a net charge on the particle surface and/or charges associated with the polymer attached to the surface (i.e. through an attached polyelectrolyte). Electrosteric stabilization is common in biological systems.

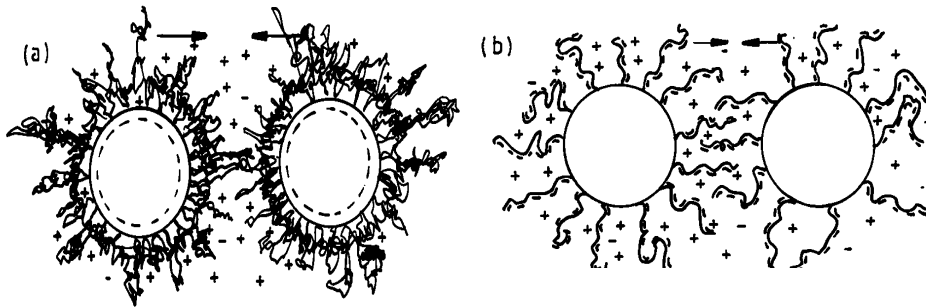


Figure 5. Diagrammatic representation of electrosteric stabilization: a) charged particles with ionic polymers; b) polyelectrolytes attached to uncharged particles.

Table 1. Major ceramic forming techniques

Pressing	Fugitive-mold casting
Uniaxial	Gel casting
Isostatic	Electrophoretic deposition
Hot pressing*	Tape casting
Hot isostatic pressing*	Doctor blade
Slip casting	Waterfall
Drain casting	Plastic forming
Solid casting	Extrusion
Vacuum casting	Roll forming
Pressure casting	Injection molding
Centrifugal casting	Compression molding

Reed, J.S. (1995). *Principles of Ceramic Processing* (2nd Ed.) New York: John Wiley & Sons, Inc.

Lee, J.K., Ko, J., and Kim Y.S. (2017). *Minerals*, 7 (165), 1-11.

Jain S.K. and Shailesh K. Jain. (1986). *Conceptual Chemistry Volume-I For Class XII*, New Delhi: S. Chand Company Pvt. Ltd.


Napper, D.H. (1983). *Polymeric Stabilization of Colloidal Dispersions*. London: Academic Press.

<http://polymerdatabase.com/polymer%20physics/Viscosity2.html>

<https://polymerdatabase.com/polymer%20physics/Viscosity3.html>

5. Content of the report



 Please interpret the experiment results.

6. Questions

1. What are the critical parameters of slip casting? Explain briefly the effect of each parameter on the process and parameters.
2. What are the advantages of gypsum as mold material?

EXPERIMENT 3 (MLZ331) TILE PRODUCTION

1. Objective of the Experiment

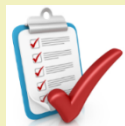
To see differences between wall, floor and porcelain tiles with respect to production and properties

2. What you should know before the experiment?



You should know;

- 📖 What are the steps of tile processing?
- 📖 Which raw materials are used for tile production?
- 📖 What are the differences between wall, floor and porcelain tiles?
- 📖 What are the roles of different raw materials?
- 📖 What are the typical properties of different types of tiles?
- 📖 What are the effects of processing parameters?
- 📖 How to do batch calculations?
- 📖 Which quality control methods are applied to finished product?
- 📖 Which parameters effect the quality of the final product?
- 📖 How to calculate percent shrinkage?
- 📖 What is liter weight?

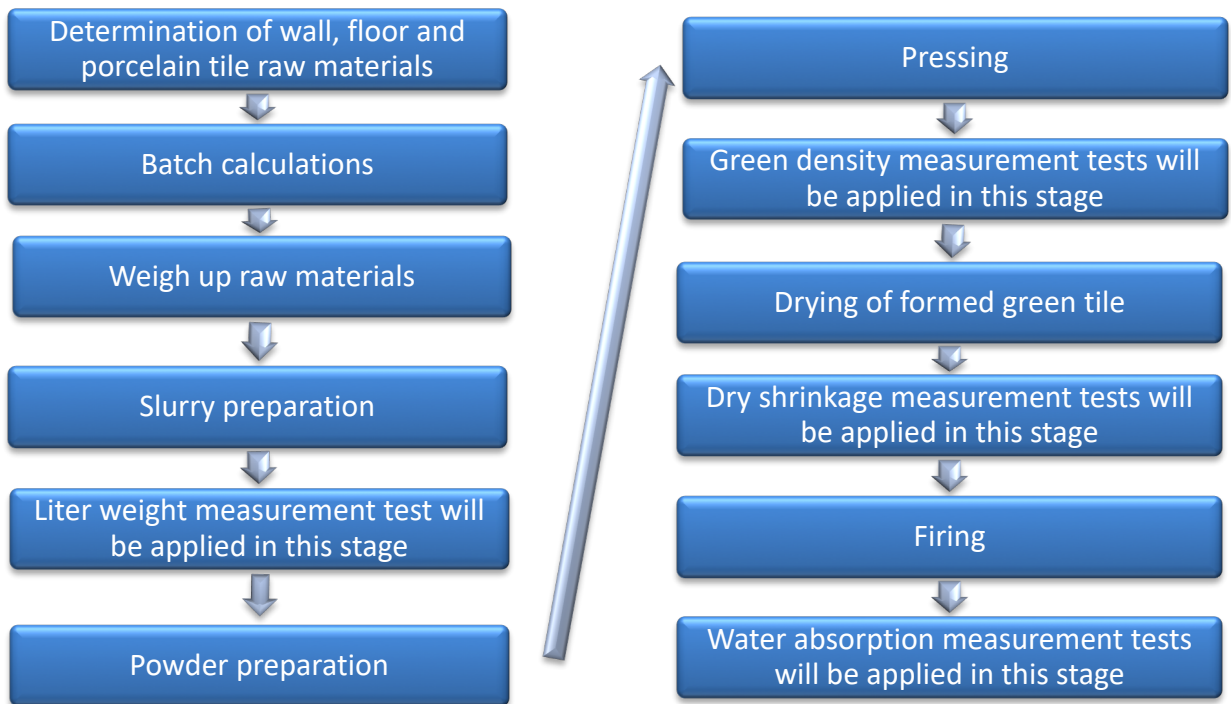


3. What will you learn during the experiment?

You will learn;

- ✍ Why different raw materials are used for different types of tiles
 - ✍ What the importance of processing conditions is
 - ✍ How to measure water absorption of tile
- ✍ Why water absorption and shrinkage values of floor, wall and porcelain tile are different
- ✍ Why different processing conditions are applied for different types of tiles
 - ✍ How to do batch calculations

4. Schematic view of experimental procedure



5. The formulations of raw materials

FLOOR TILE		PORCELAIN TILE		WALL TILE	
Raw materials	Weight %	Raw Materials	Weight %	Raw Materials	Weight %
Clay	30	Kaolin	20	Clay	50
Kaolin	25	Clay	10	Kaolin	30
Na-Feldspar	20	Ukraine Clay	20	Calcite	10
Pegmatite	25	Na-Feldspar	50	Pegmatite	10

5. Equipments and materials

- 📌 Tile raw materials
- 📌 Laboratory scale
- 📌 Ball mill
- 📌 Uniaxial press
- 📌 Fast sintering furnace

6. Important points / hints for the equipments and/or results obtained from the analyses

- 🗨️ Weighting accuracy of raw materials is important
- 🗨️ Differences in parameters of grinding of the raw materials and slurry preparation
 - 🗨️ Sintering temperature differences
 - 🗨️ Liter weight
 - 🗨️ Differences in shrinkage values
 - 🗨️ Differences in water absorption

7. Content of the report



- 📄 Please write all information, measurements and etc. that you obtain during the experiment
- 📄 Please show the calculations of average shrinkage values after drying and sintering for all samples and explain why there is a difference in shrinkages between different types of tiles
- 📄 Please write the water absorption conditions and also show the calculations of water absorption and explain why there is a water absorption difference between different types of tiles



8. Questions

1. Write the differences between wall, floor and porcelain tiles on the basis of raw materials and physical properties.
2. What is the purpose of using kaolin in wall, floor and porcelain tiles?

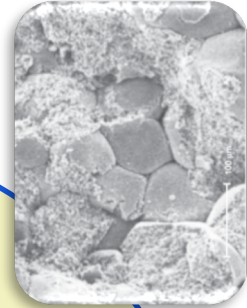
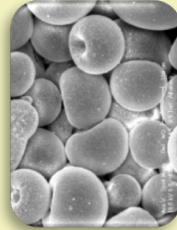
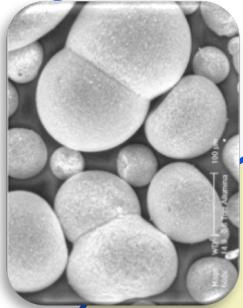


1. Objective of the Experiment

- To show how to form ceramic objects by pressing various types of powders
- To show the pressure and thickness effect on green density

2. What you should know before the experiment?

You should know;



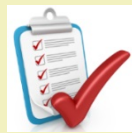
📖 All the "you should know & what you will learn" sections in Exp. 1.

📖 What are the stages of pressing?

📖 What is the importance of powder characteristic?

📖 What are the parameters that should be considered during pressing?

📖 What are the defects that occurred during and after pressing?



3. What will you learn during the experiment?

You will learn;

✂ How to form ceramic powders by using dry pressing?

✂ How the thickness affects the green density?

✂ How the pressure affects the green density?

✂ How the defects can occur during and after pressing?

✂ How to control the compaction defects?

4. Schematic view of experimental procedure

Weighing the powder



Filling the die



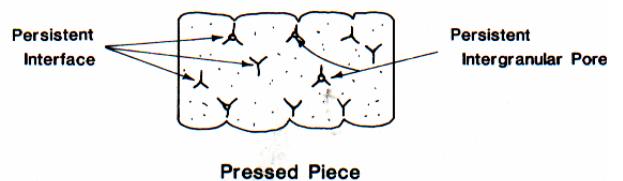
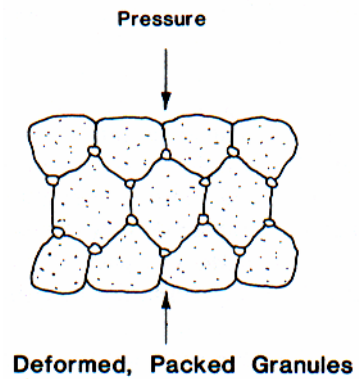
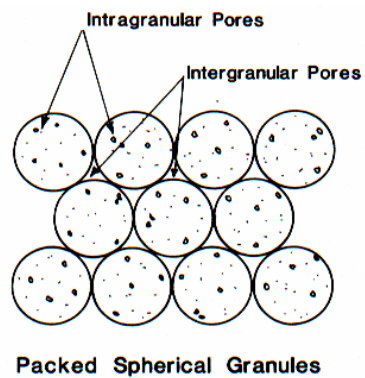
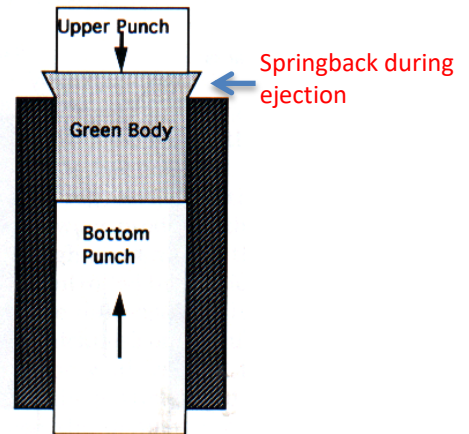
Pressing under designated pressure



Ejecting the sample out of the die



Measure and record weight and dimensions of the sample



5. Equipments and materials (if necessary user manuals)

- 📌 Ceramic powder (granulated and non-granulated),
- 📌 Pressing dies,
- 📌 Mechanical press.

6. Important points / hints for the equipments and/or results obtained from the analyses

- 🗨️ Good powder flow is essential for reproducible volumetric filling, a uniform density of the fill and a rapid pressing rate.
- 🗨️ Hard granule difficult to change shape, causing residual pore, thus lowering product strength
- 🗨️ The compact must survive ejection and handling without failure and should be free of defects.
- 🗨️ Using lubricant during pressing is important to be aware of frictional forces.
- 🗨️ To minimize defect formation, some pressure is kept during ejection process.
- 🗨️ Air problem can be minimized by de-airing before compression.

7. Content of the report



- 📄 Please write all information that you learn during the experiment
- 📄 Please write all the data that you note/measure/calculate during the experiments
- 📄 For each pressing condition the green density vs. pressure graph will be plotted and then pressure effect on the green density will be evaluated.
- 📄 The green density vs. pressure results will be plotted. The results obtained from the thickness effect will be compared and granulation effect on compaction behavior will be evaluated.
- 📄 Please answer the Questions stated in section 8 in a separate paper



8. Questions

1. What is the importance of good flowability of powder for dry pressing?
2. What are the causes of density gradient in the pressed compact? Explain the relationship between pressing defects and density gradient.



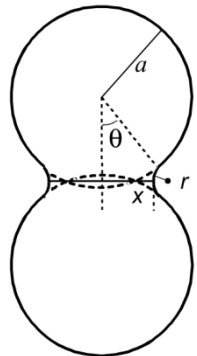
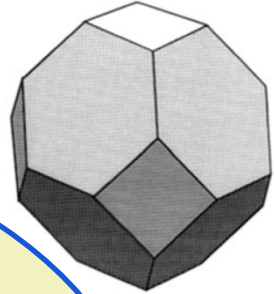
1. Objective of the Experiment

- To learn sintering process of ceramic materials.
- To evaluate sintering graphs
- To measure the density of sintered ceramic bodies

2. What you should know before the experiment?

You should know;

- 📖 All the "you should know & what will you learn" sections in Experiment 6.
- 📖 What is sintering?
- 📖 What are Solid State Sintering, Liquid Phase Sintering, Viscous Flow Sintering and Pressure Assisted Sintering?
- 📖 What types of furnaces are used for sintering ceramics?
- 📖 What are the basic components of a furnace?
- 📖 What are density and porosity?
- 📖 What is Archimede's principle?

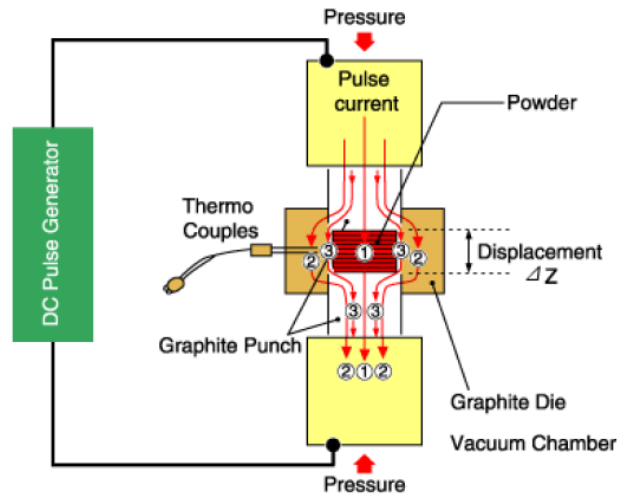
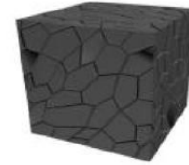
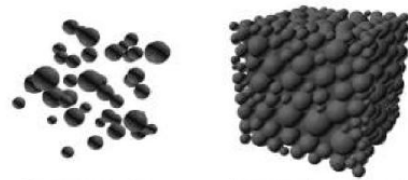
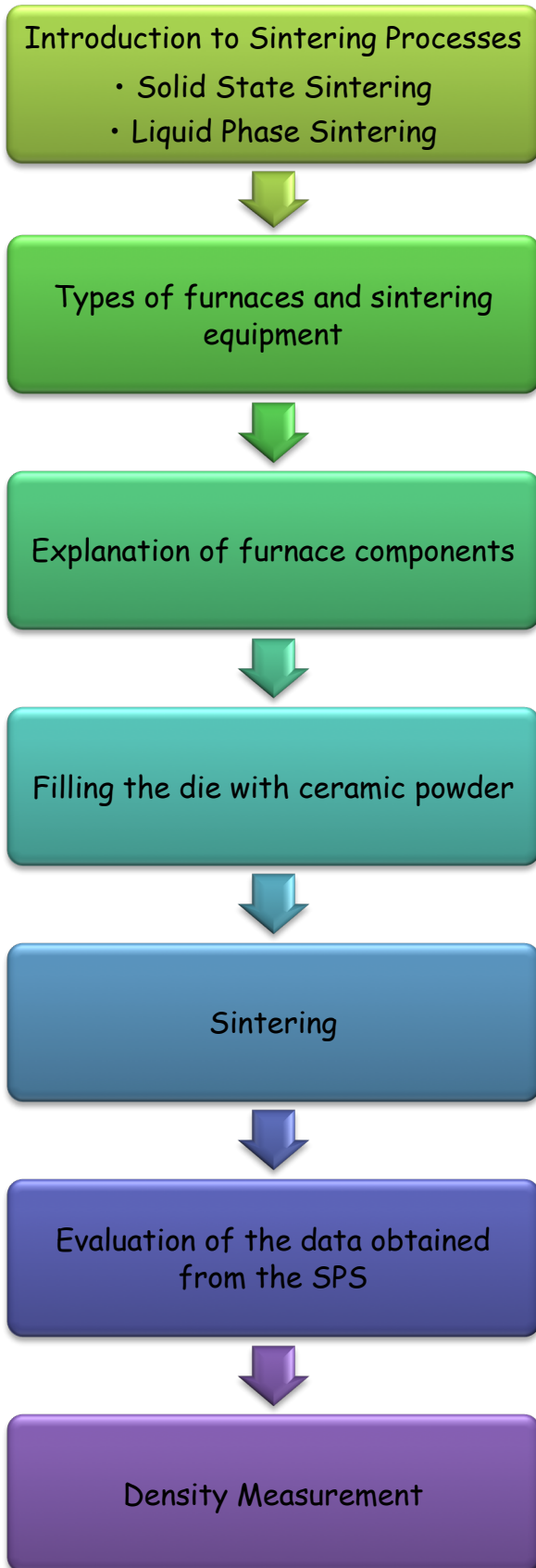


3. What will you learn during the experiment?

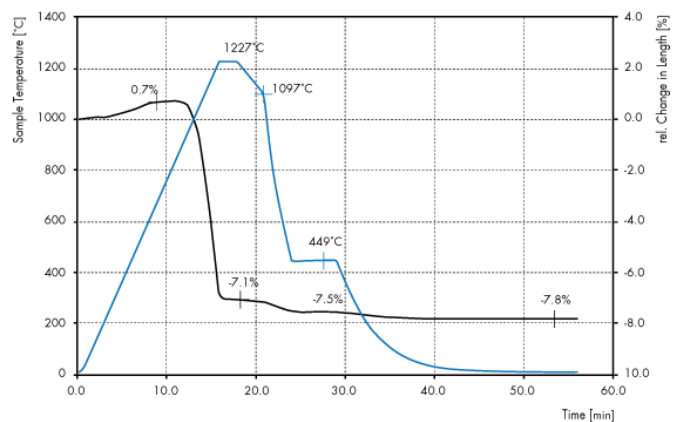
You will learn;

- ✂ How to estimate the sintering profile of a ceramic body.
- ✂ How to sinter ceramic powders to a dense body.
- ✂ The sintering mechanisms and material transport mechanisms during sintering processes.
- ✂ How sintering parameters affect properties of the final product.
- ✂ How to measure the density of sintered product.
- ✂ How to calculate the density of porous materials.

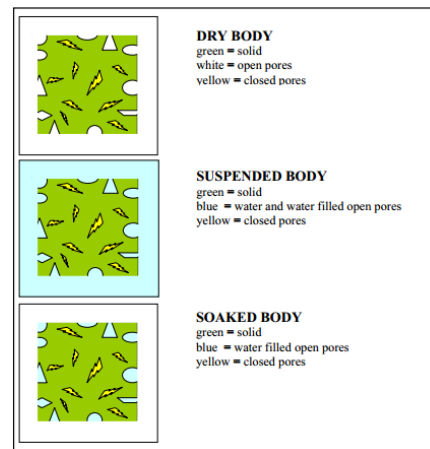
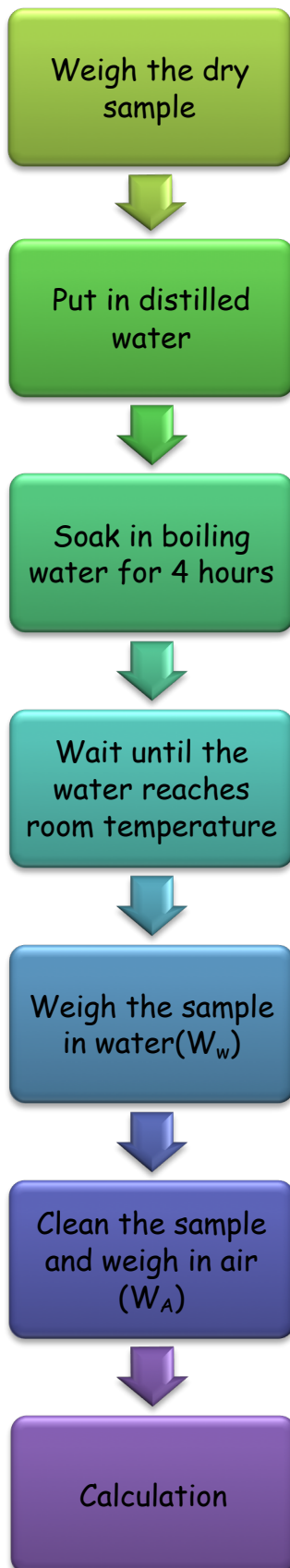
4. Schematic view of experimental procedure



Ceramic (fast firing process)




5. Density Measurement



- Bulk Density (BD) = $\frac{W_{dry}}{W_A - W_w} \times \rho_{water}$
- Water absorption ($\%WA$) = $\frac{W_A - W_{dry}}{W_{dry}} \times 100$
- Apparent Porosity ($\%AP$) = $\frac{W_A - W_{dry}}{W_A - W_w} \times 100$
- Apparent Solid Density (ASD) = $\frac{W_{dry}}{W_{dry} - W_w}$


6.Equipments and materials (if necessary user manuals)

- 📌 Appropriate ceramic powder
- 📌 Spark plasma sintering furnace
- 📌 Precision scale and Archimedes density equipment



7.Important points / hints for the equipments and/or results obtained from the analyses

- 🗨️ Correct preparation of the die.
- 🗨️ Sintering process parameters.
- 🗨️ Reasonable interpretation of the sintering graphs.
- 🗨️ Density evaluation.



8.Content of the report

- 📄 Content of the report will be given to you at the lab course.



9. Questions

1. Give the definitions of the words given below
(i) Sintering (ii) Densification (iii) GPS (iv) SPS
2. Explain the Archimede's principle by drawing.



1. Objective of the Experiment

To show how to prepare and apply glaze and explain the most important parameters in terms of obtaining decent glazes.

2. What you should know before the experiment?

You should know;



📖 All the "you should know & what will you learn" sections in Exp. 4.

📖 What is glaze?

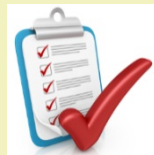
📖 Why are glaze coatings applied to products?

📖 What are the most important parameters in terms of obtaining decent glazes?

📖 What are the different methods of glaze application?

📖 Why is the rheology of the glaze important?

📖 What does deflocculant provide?



3. What will you learn during the experiment?

You will learn;

✍️ Why is frit used in glazes?

✍️ What are the differences between fritted and unfritted glaze?

✍️ What properties are supplied to products by applying glaze?

✍️ What are the types of glazes?

✍️ Which raw materials are used for preparing glaze and what are their functions?

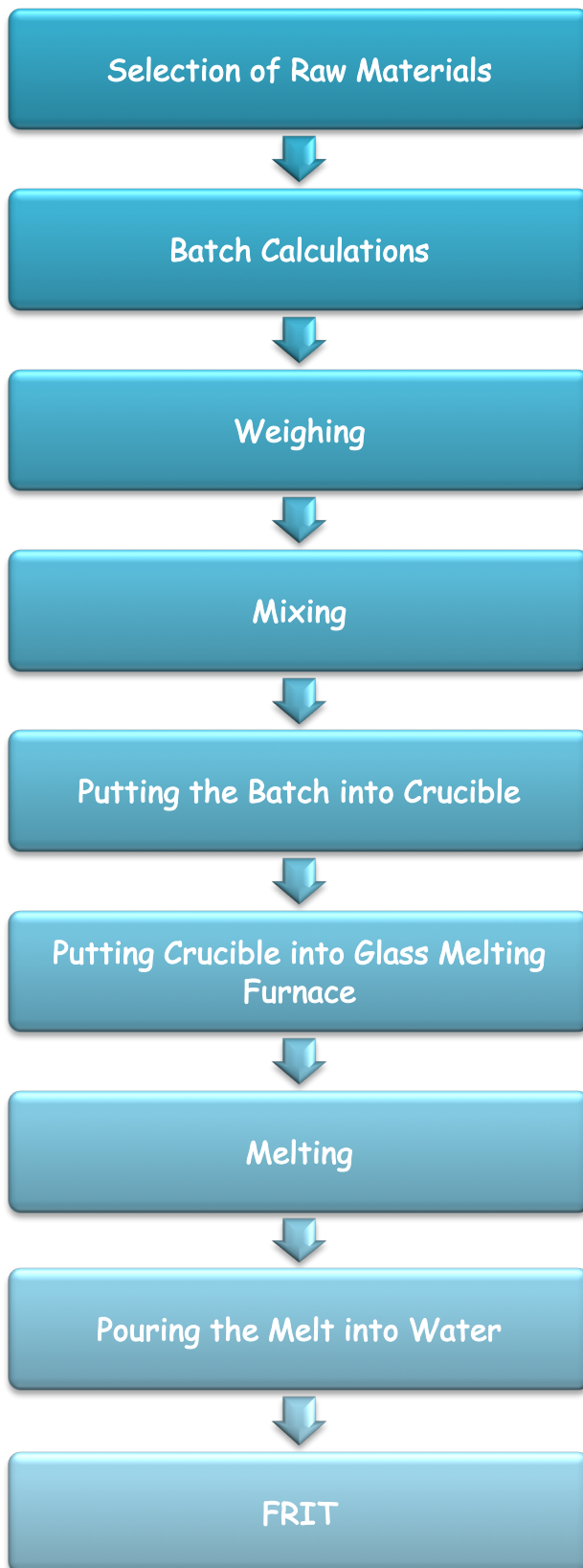
✍️ How do we apply glaze on the products?

✍️ Why do we use CMC and STPP?

✍️ How do we determine the firing temperature for a glaze?

✍️ What are the common defects in glazed products?

4.Schematic view of experimental procedure



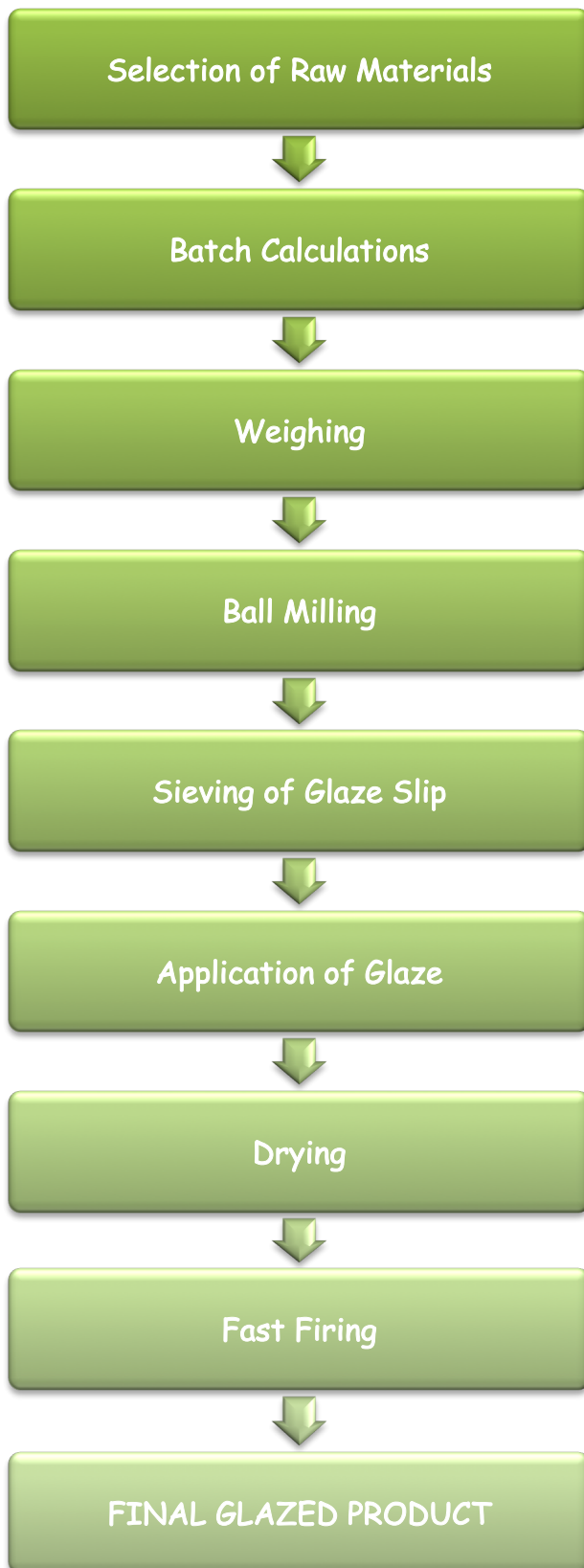
Glaze Recipe

Raw Materials	Weight %
Boric acid	5.46
Zinc oxide	6.54
Calcite	12.64
Alumina	16.44
Quartz	34.42
Potassium nitrate	3.45
Sodium feldspar	15.35
Barium carbonate	5.71

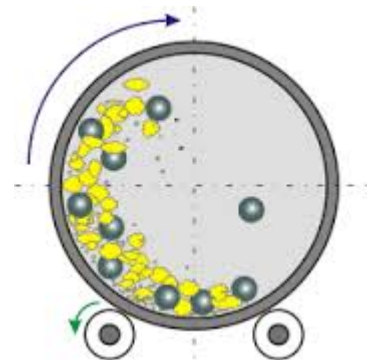
Frit



4.Schematic view of experimental procedure



Milling



Glaze Application



5. Equipments and materials (if necessary user manuals)

For Fritting;

- 📌 raw materials for various frits
- 📌 bottom loading glass melting furnace
- 📌 porcelain or platinum crucible

For Glazing;

- 📌 grinding mills and media
- 📌 spraying, dipping and pulling apparatus
- 📌 fast firing furnace
- 📌 drying oven

6. Important points / hints for the equipments and/or results obtained from the analyses

- 🗨️ criteria for materials' selection
 - 🗨️ milling parameters
 - 🗨️ application techniques
 - 🗨️ firing
- 🗨️ body-glaze interactions

7. Content of the report



- 📄 Please write all information that you learn during the experiment.
- 📄 Please interpret the experiment results according to list of section 3 and schematic view of experimental procedure in section 4.
- 📄 Please answer the Questions stated in section 8 in a separate paper.



8. Questions

1. Why do you apply glaze coatings on to substrates?
2. What are the common defects in glazed products? Please explain.



1. Objective of the Experiment

- The objective is to learn thin film deposition processes and the main important parameters effecting thin film properties
- To determinate thin film properties (e.g. thickness, surface morphology, electrical conductivity) via characterization techniques as Atomic Force Microscopy (AFM), X-Ray Reflectometry and Four Point Probe (FPP)

2.What you should know before the experiment?

You should know;

- ✚ *All the “you should know and you will learn” sections in this Exp.*
- ✚ *What are the deposition methods and what are they used for?*
- ✚ *What is vacuum and why it is necessary for thin film deposition?*
- ✚ *What is the importance of growth rate(i.e. deposition rate) in thin film deposition process?*
- ✚ *What is “Island, Layer-by-Layer Growth and Mixed Growth?*
- ✚ *What are the thickness and average grain size analysis methods and their basis working principles? What kind of information could be obtained from these techniques?*

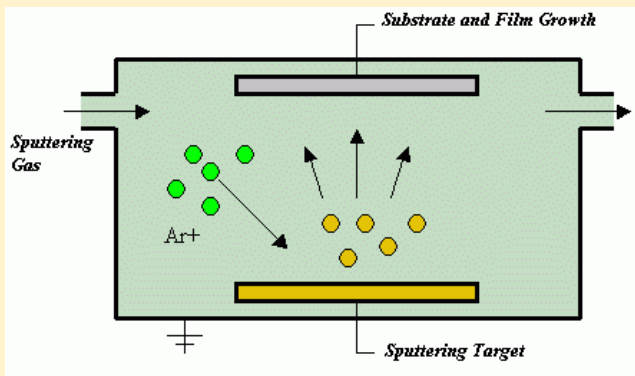
3.What will you learn during the experiment?

You will learn;

- ✚ *How to prepare metallic and metal-oxide (ceramic) thin films?*
- ✚ *How to determine the surface morphology by AFM?*
- ✚ *How to measure deposition rate and thickness of the film-coating by using X-Ray Reflectometer method?*
- ✚ *How to measure TCR via FPP*



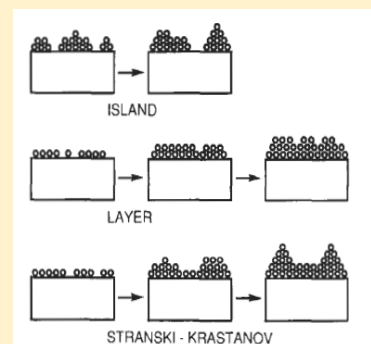
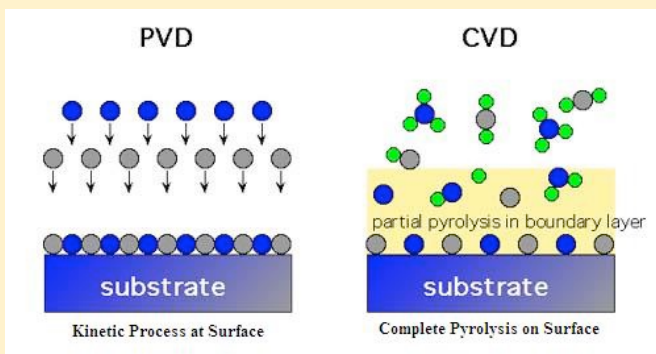
Thin films are fabricated by the deposition of individual atoms on a substrate. A thin film is defined as a low-dimensional material created by condensing, one-by-one, atomic/molecular/ionic species of matter. The thickness is typically less than several microns. Thin films differ from thick films. A thick film is defined as a low-dimensional material created by thinning a three-dimensional material or assembling large clusters/aggregates/grains of atomic/molecular/ionic species.



In all deposition techniques and application of thin films the vacuum technologies are imperative. Any thin-film deposition process involves three main steps: 1. Production of the appropriate atomic, molecular, or ionic species. 2. Transport of these species to the substrate through a medium. 3. Condensation on the substrate, either directly or via a chemical and/or electrochemical reaction, to form a solid deposit.

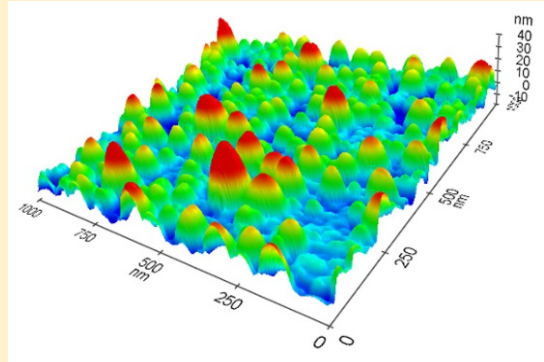
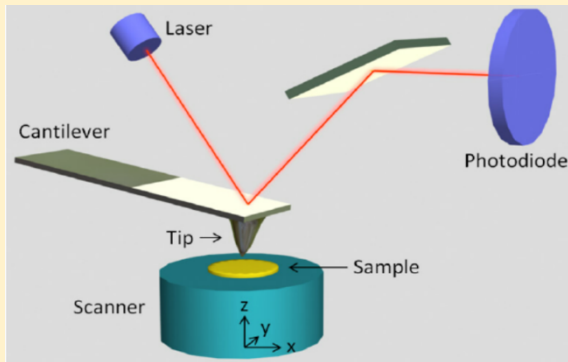
The Steps in Film Formation: 1. Thermal accommodation; 2. Binding; 3. Surface diffusion; 4. Nucleation; 5. Growth; 6. Coalescence; 7. Continued growth. Depending on the thermodynamic parameters of the deposit and the substrate surface, the initial nucleation and growth stages (4 and 5) of the many observations of film formation have pointed to three basic growth modes may be described as (a) island type, called Volmer-Weber type, (b) layer type, called Frank-van der Merwe type, and (c) mixed type, called Stranski-Krastanov type.

Typical deposition processes are physical and chemical. The physical process is composed of the physical vapour deposition (PVD) processes, and the chemical processes are composed of the chemical vapour deposition (CVD) process.



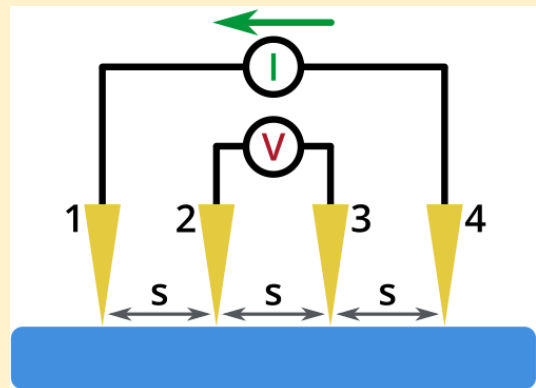
Experimental techniques and applications associated with determination of “film thickness”, “film surface morphology and structure”, “film and surface composition”, “electrical properties of films”.

The Atomic Force Microscope (AFM), which is sometimes called the Scanning Force Microscope (SFM), is based on the forces experienced by a probe as it approaches a surface to within a few angstroms.




XRR is a non-destructive and non-contact technique for thickness determination between 2-200 nm with a precision of about 1-3Å. In addition to thickness determination, this technique is also employed for the determination of density and roughness of films and also multilayers with a high precision.

The purpose of the 4-point probe is to measure the resistivity or average resistance of a thin layer or sheet or any semiconductor material by passing current through the outside two points of the probe and measuring the voltage across the inside two points.



4. Equipments and Materials

-  Chemicals
-  Substrates
-  Targets
-  Sputtering system
-  Atomic Force Microscopy
-  X-Ray Reflectometry
-  Four Point Probe

5. Schematic View of the Experimental Procedure

Preparation of substrates by using Dicer, chemicals, ultrasonic bath and, pure (%99.9995) N₂ gas



Loading of the Substrates



Thin Film Deposition by Magnetron Sputtering

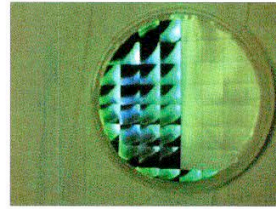


Morphological analysis by AFM



Thickness, roughness and density analysis by X-Ray Reflectometer

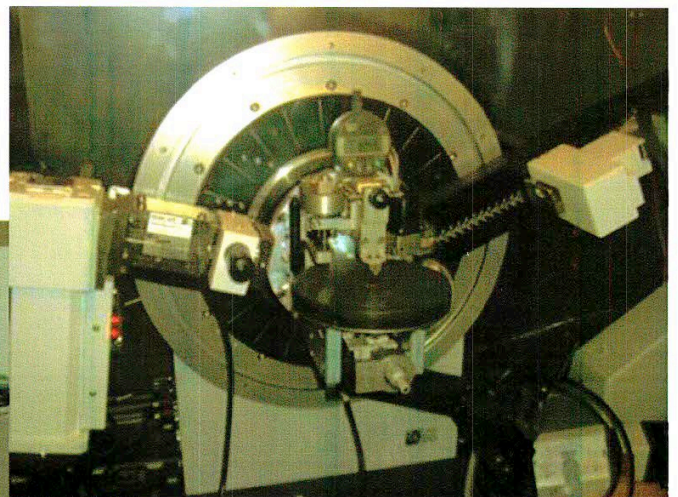
Dicing of 3 inch Substrate (Dicer),
Substrate Cleaning



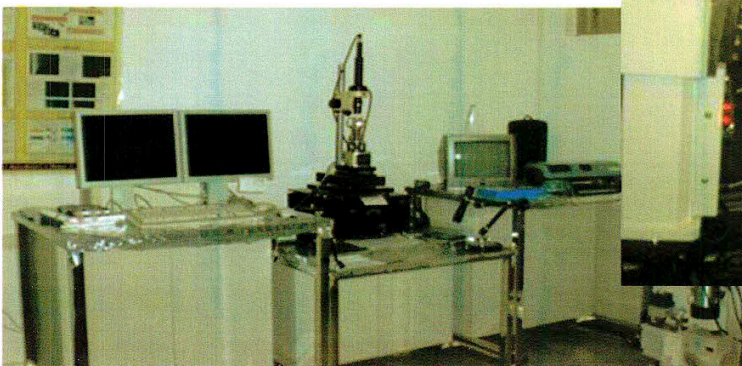
Sputtering System



X-Ray Reflectometer



Atomic Force Microscope



6. Content of the Report

- I. Briefly describe the AFM contact mode, AFM non-contact mode and AFM tapping mode.
 - Assume that you have a soft sample (e.g. biological sample) Which mode will you prefer? Explain.
- II. Plot the $\ln R - T$ (Resistance to Temperature) graph using the given table and calculate the TCR.
 - Decide the best substrate for the application that you have chosen.

Content of the report may change during the course !

7. Questions

1. What is thin film? What are the three basic growth modes observed during thin film formation? (please explain by drawing figure.)
2. Explain the working principle of the magnetron sputtering technique (please explain by drawing figure.). What are the important parameters in thin film deposition with magnetron sputtering technique?