



Erlangen

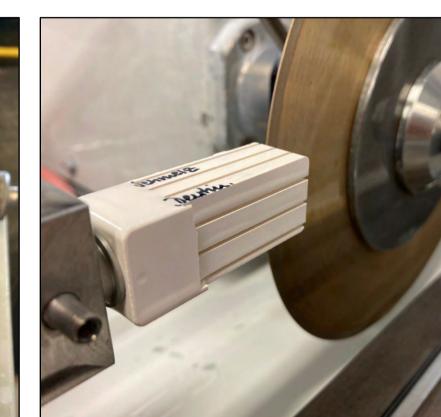
# Sample Preparation



#### Serial Sectioning

- For the measurements of physical and mechanical properties, specimens must comply to specific geometries defined by the literature or testing standards.
- Commonly, materials are produced in larger volumes or supplied by manufacturers in pre-fabricated shapes that must be processed to the desired size and geometry.
- Serial sectioning is done with the aid of manual or automatic saws that cut materials with low-speed diamond-coated copper discs, whether dry or under water lubrication.





- (Left): Sectioning of square plates of glass using a manual saw.
- (Right): Sectioning of presintered zirconia blocks in beams mounted to a cubic specimen holder to allow 90° rotation in an automatic sawing machine under water lubrication.



#### Machining

- Machining is the process of shaping ceramic materials via a subtractive route, following the Computer-Aided-Design/Computer-Aided-Machining (CAD/CAM) technology.
- Burs of several shapes and diamond sizes are mounted in CAM machines that work around 3 to 5 axes to grind pre-fabricated blocks of blanks to the desired shape.
- CAM machines are used to grind commercial materials to anatomical prosthetic shapes for bench-testing or simplified geometries using exported STL files.





- ▶ (Left): Machined block of a commercial lithium disilicate material into a crown shape.
- ▶ (Right): Zirconia block mounted on a 3-axis CAM unit having a doublespindle.



#### Surface Grinding and Polishing

- The procedure of grinding is meant to remove large amounts of material in the z-plane while maintaining the grinded surface parallel to the lower surface plane.
- Rotary grinding machines use diamond wheels of different grades employed depending on the amount of subtraction, type of material and severity of surface damage.
- Manual or automatic polishing machines use SiC papers or discs followed by cloths used in conjunction of oxide suspensions of varying particle sizes.





- ► (Left): Square glass plate specimens mounted on a holder for grinding and subsequent polishing.
- ► (Right): Automatic polishing machine with holder on a rotating SiC disc.



## Acid Etching

- Etching procedures are done for two purposes: (i) for revealing microstructural elements in glass-ceramics and (ii) cleaning platinum crucibles used for glass melting.
- Hydrofluoric (HF) acid is used to dissolve SiO<sub>2</sub>-based glasses in various concentrations, from 0.5% for surface etching up to 40% for cleaning platinum crucibles.
- In high concentrations, HF must be stored safely in Polytetrafluoroethylene or Polypropylene containers inside fume hoods with constant vapour suction.





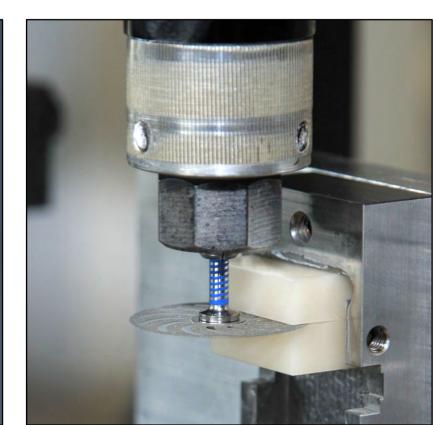
- ▶ (Left): monolithic PP container for 40% HF storage for cleaning of glass melting crucibles.
- (Right): Low (5%) concentration HF in gel form for etching of ceramic surfaces.



### Moulding / Shaping / Notching

- In order to produce samples with a desired geometry, specimens can be shaped by moulding of viscous materials or shaping of solids by cutting and grinding.
- Resin composites are inserted in sliced moulds made of teflon or tungsten carbide having plasma eroded surface for perfect finish.
- Notching is the procedure of producing a starter pre-crack or to constrain the crosssection during the fabrication of fracture toughness specimens.





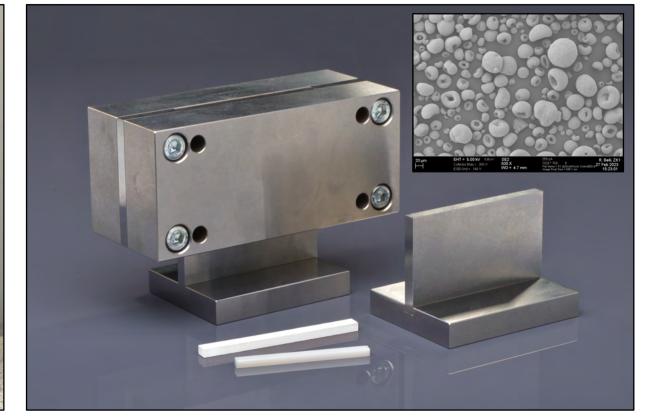
- ▶ (Left): Procedure for the production of a resin composite beam by moulding in a split WC mould.
- (Right): Notching of a ceramic block using a diamond disc for the production of a pre-crack in CT specimens.



## Powder Compaction

- Oxide ceramics are produced by compaction of powder granulates under uniaxial pressing followed by cold isostatic pressing.
- Powder compacts are called green-bodies that will be subsequently sintered aiming for increasing densification and grain growth.
- For the production of discs and beam-shaped specimens, hardened metallic forms are used under an hydraulic press to up to 100 MPa.





- ▶ (Left): Hydraulic press used to uniaxially press beam or discshaped (photo) green-bodies.
- (Right):Metallic form used to press beamshaped specimens. Inset: powder granulate of zirconia particles.