Glass Handling
Best Practices for Glass Primary Containers

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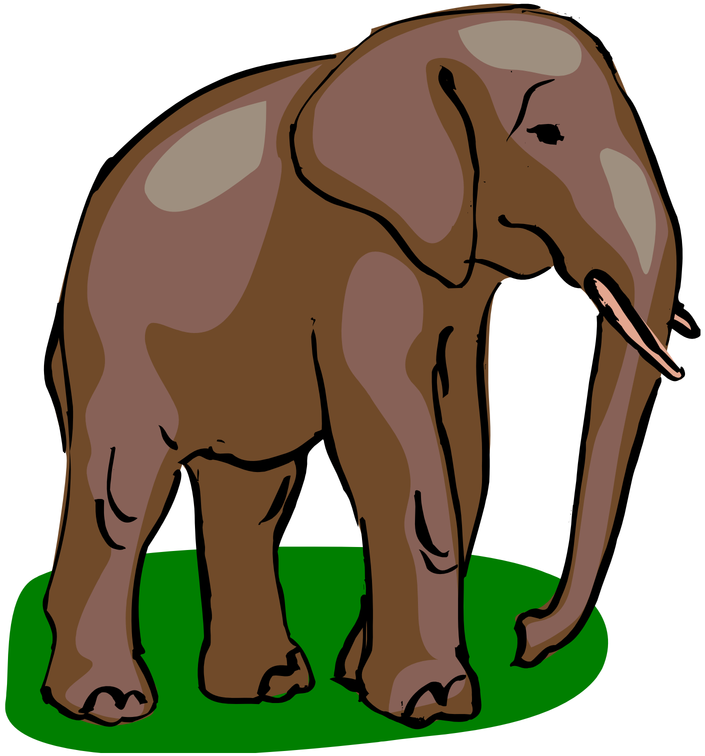
Outline

- Glass Breakage – Fundamentals
- Assessment of flaws
- Fractography – Fundamentals

Or – what does glass have in common with an elephant?



Root cause for glass breakage

- Simultaneous presence of
  - Flaw (critical in terms of mechanical strength)
  - Mechanical load (tensile stress) at flaw
- Interaction of critical flaw and mechanical load (“stress intensity”) reaches critical value (“breakage resistance”)

Glass breakage: *surface flaw × tensile load ≥ breakage resistance*

stress intensity

Different cases

- No breakage, if no or only one factor is present
- Flaw and mechanical load occur simultaneously
  - Impact
- Flaw is created prior mechanical load
  - Depyrogenization/heat sterilization
  - Lyophilization/freeze drying
  - Auto-injector
- Flaw is introduced while mechanical load is already present
  - Residual stresses
  - Constant internal pressure

Definition of "flaw"

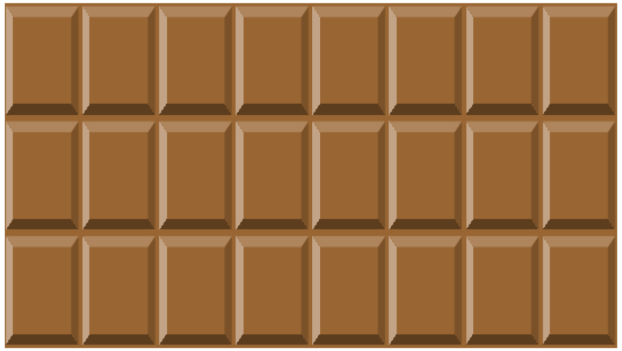
- Any type of discontinuity within the isotropic, monolithic structure of the glass (including the surface) can act as flaw
  - Foreign material
  - Voids
  - Surface irregularities
- Discontinuities act as concentrators for mechanical stresses
- Also variations in geometry
- Size (dimension) *and* shape (geometry) of discontinuity affect criticality
  - Large flaws can exhibit low criticality
  - Small flaws can exhibit high criticality

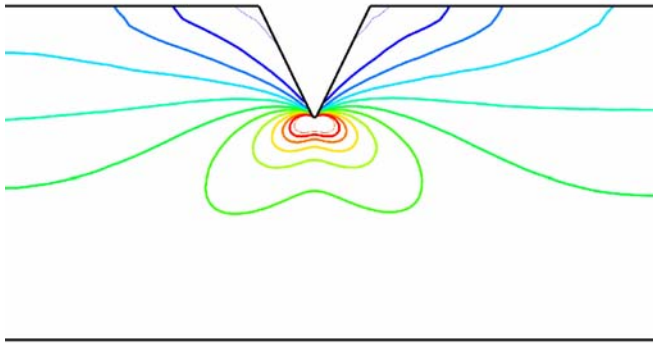
Any type of discontinuity within the isotropic monolithic structure of a glass (including the surface) can act as flaw and become critical in terms of strength

- Criticality affected by size and shape

Intensification of mechanical stresses

- Discontinuities act as concentrators for mechanical stresses
- Example: Bar of chocolate
  - Notches act as stress concentrators ("surface flaws")
  - Contribution of notches to stress intensity factor ≈2 higher than for plane bar
  - Lower tensile loads (≈1/2) for breakage required



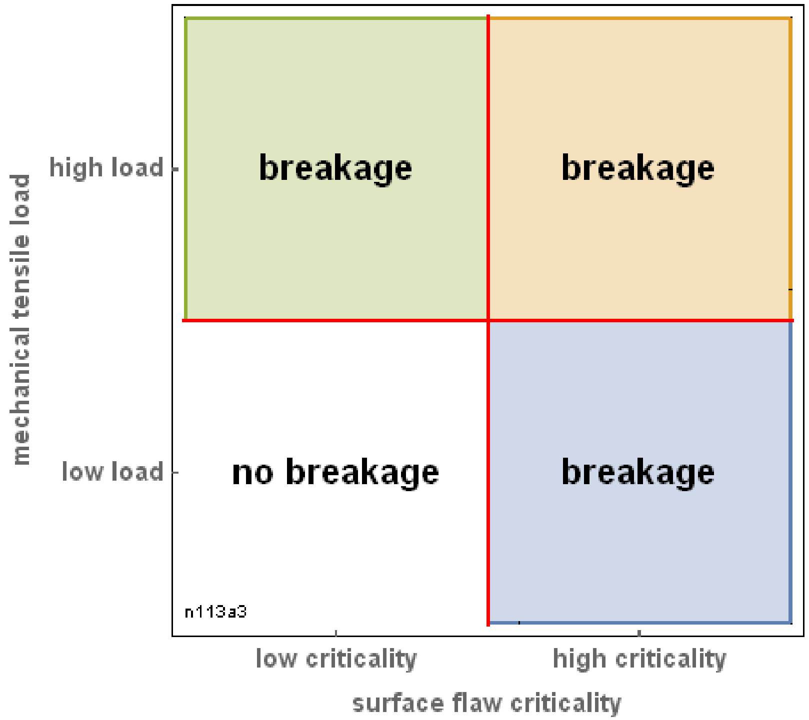


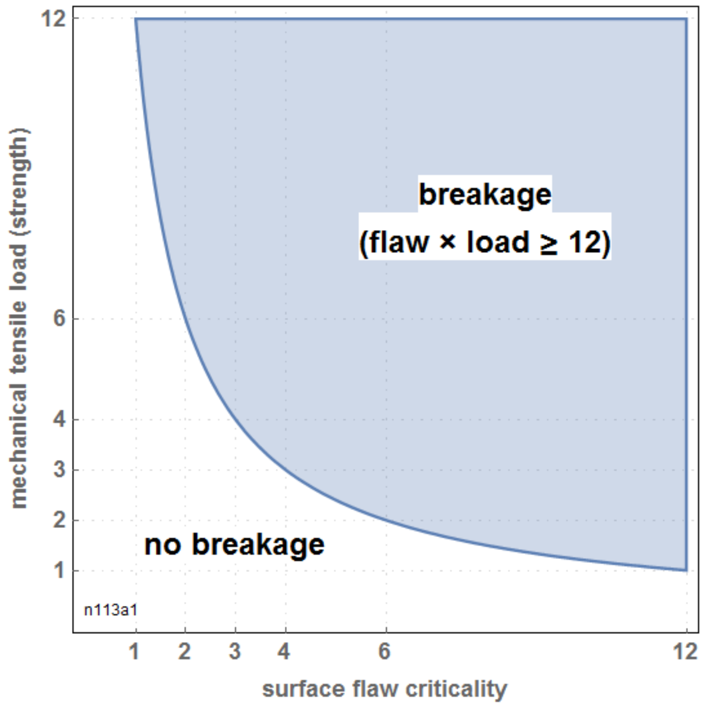
	surface flaw	×	tensile load	≥	breakage resistance
plane	1	×	4	≥	4
notched	2	×	?	≥	4

Determination of failure criteria

- Definition of strength: Mechanical resistance against breakage
  - Value/magnitude of mechanical load at which breakage occurs

Glass breakage: *surface flaw × tensile load ≥ breakage resistance*





Determination of failure criteria

- Definition of strength: Mechanical resistance against breakage
  - Value/magnitude of mechanical load at which breakage occurs

Glass breakage: stress intensity ≥ breakage resistance

- Strength depends on combination of flaw and load contribution

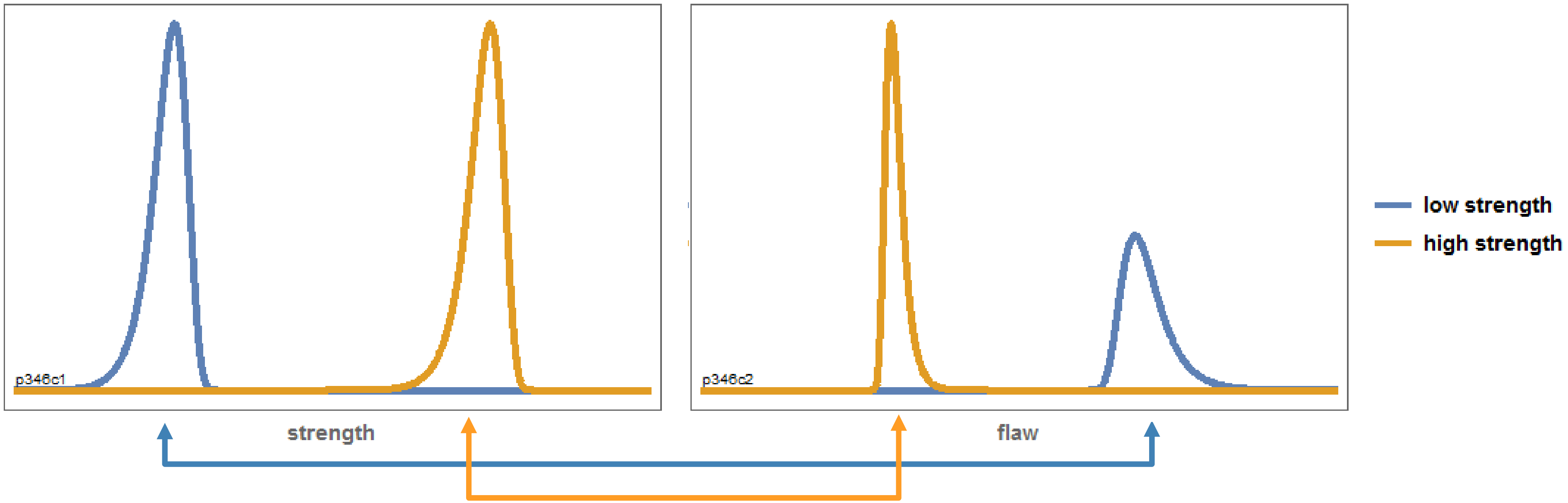
The strength of glass is *not* a material constant

The strength of glass is a projection of its surface quality

- Surface quality defined by flaw size (distribution)

Determination of failure criteria

- Flaw size distribution → strength distribution
  - Large flaws → low strength
  - Small flaws → high strength



Determination of failure criteria

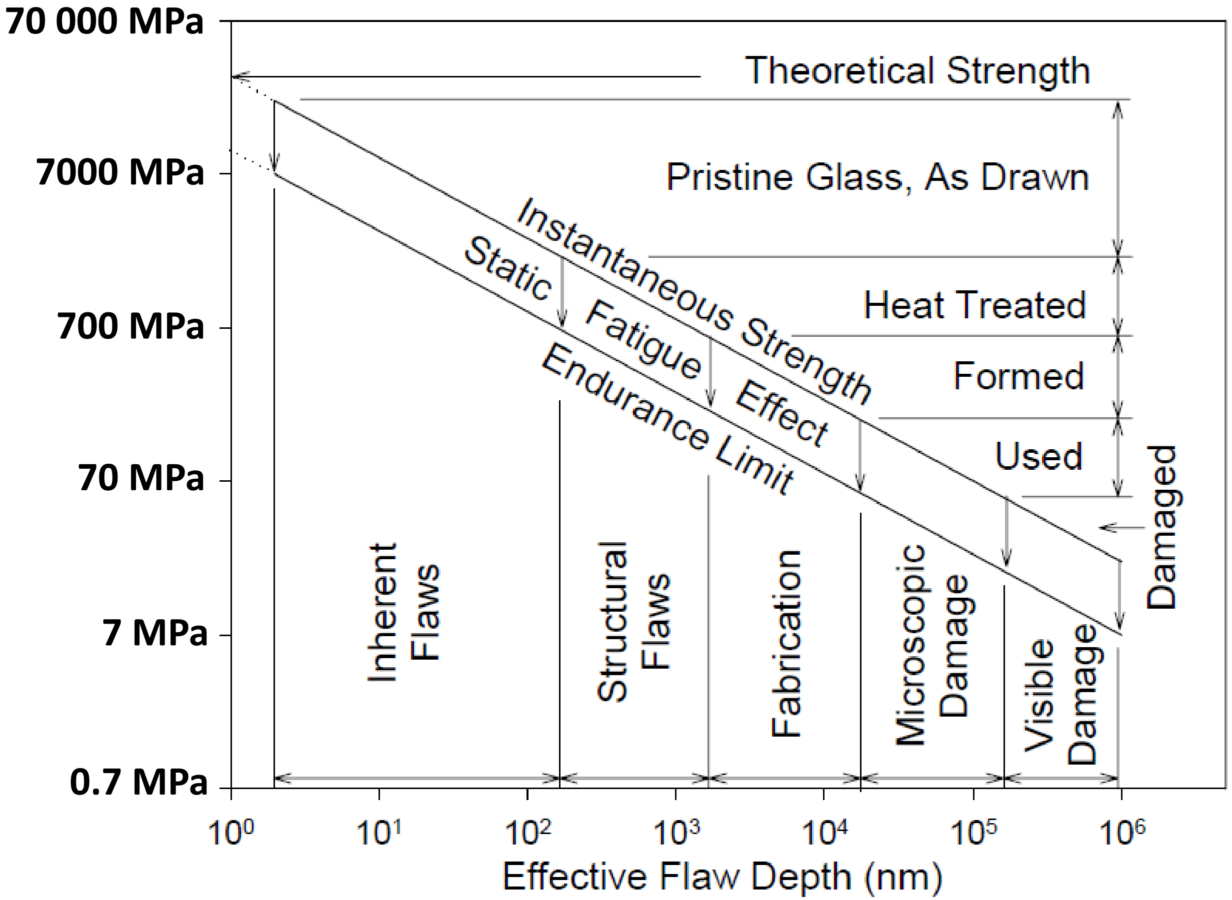
- The surface quality of glass is defined by the
    - Type(s)
    - Criticality (shape)
    - Size distribution(s)
    - Number/amount

  of surface flaws
- Every glass surface contains flaws
- A perfect glass surface without any flaws does not exist

Consequence: Flaws limit the strength of a glass solid

Reduction of strength

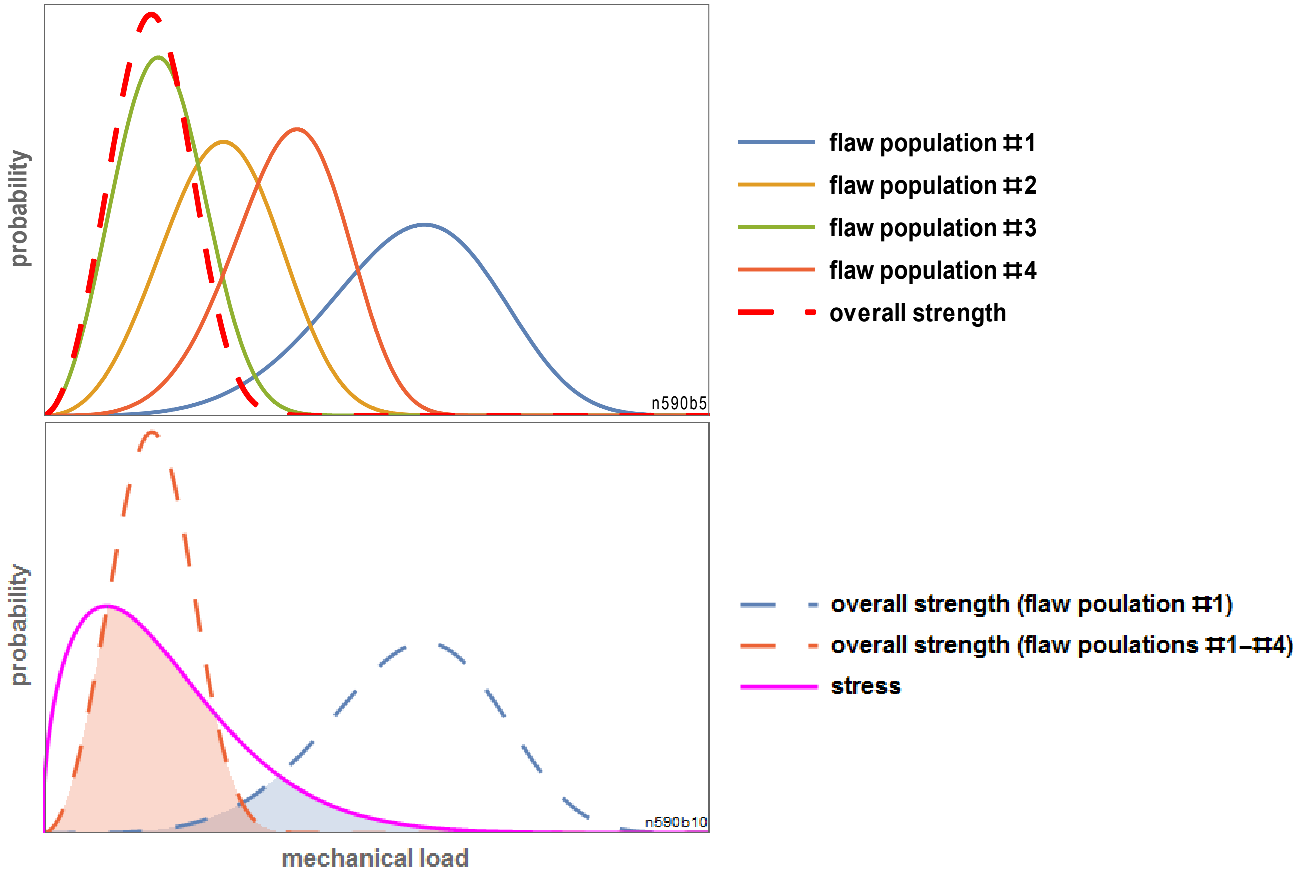
- Increase in flaw size (depth) reduces strength



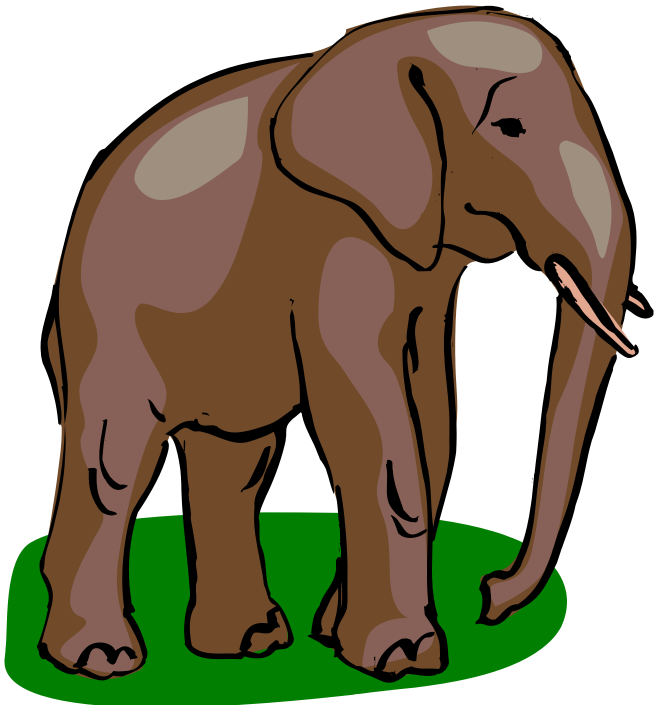
[R.E. Mould, in: Fundamental Phenomena in the Materials Sciences, ed. L.J. Bonis, J.J. Duga and J.J. Gilman, 119-149 (1967)]

Multiple flaw populations

- The strength distribution of the most severe flaw population dominates at low mechanical loads



So what does glass have in common with an elephant?



Both do not forget and do not forgive any mistreatment!

Overall summary: Treat your glass (and your elephants) right!

Assessment of flaws (in terms of breakage criticality)

- Different publishers
  - PDA Technical Report #43
  - Editio Cantor Verlag
  - Container vendors
  - Company-internal
- Defect catalogues
  - In general: No distinction between cosmetic and strength-affecting flaws
  - Characterization and assessment of flaws only by (lateral) dimensions
- Required information for assessment of criticality
  - Flaw shape/geometry, container shape/geometry → (three-dimensional) geometry information
  - Flaw dimension → flaw size (“depth”)

Assessment of flaws in terms of breakage criticality

- Are optical techniques capable to acquire information about (three-dimensional) flaw geometry and depth?
  - Manual (human eye)?
  - Automated (camera/software)?

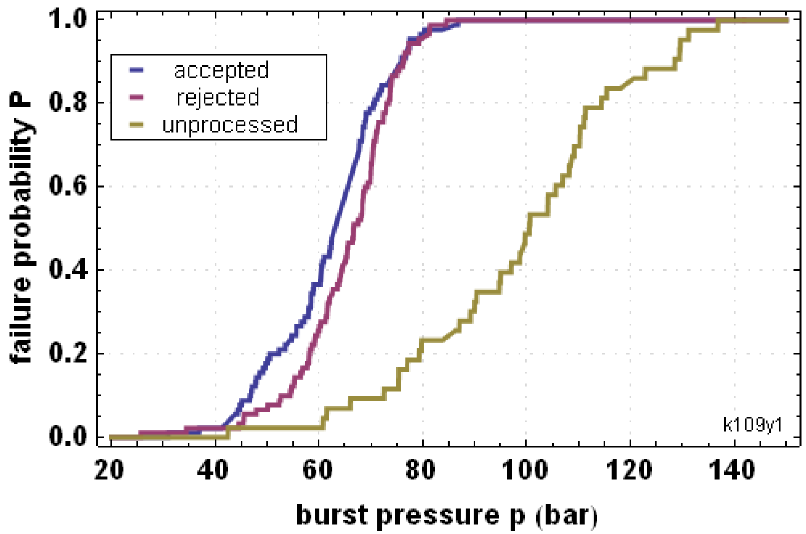
Optical inspection systems are inappropriate for an assessment of criticality

- Reliable assessment of strength-related flaws only possible via appropriate strength experiments

Only strength experiments are capable to acquire reliable information about criticality of flaws

Cosmetic versus critical flaws (example)

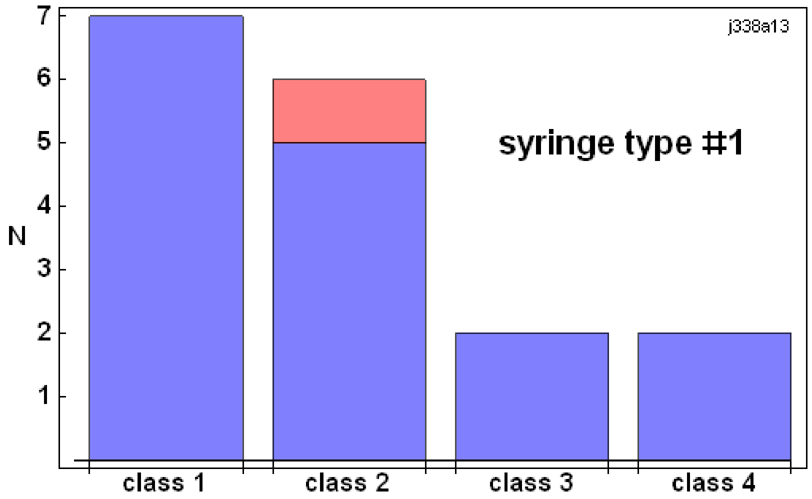
- Batch of glass vials rejected due to cosmetic flaws
- Accepted reference batch (no cosmetic flaws)
- Burst-pressure strength experiments
- Fractographic examinations (location of fracture origin)

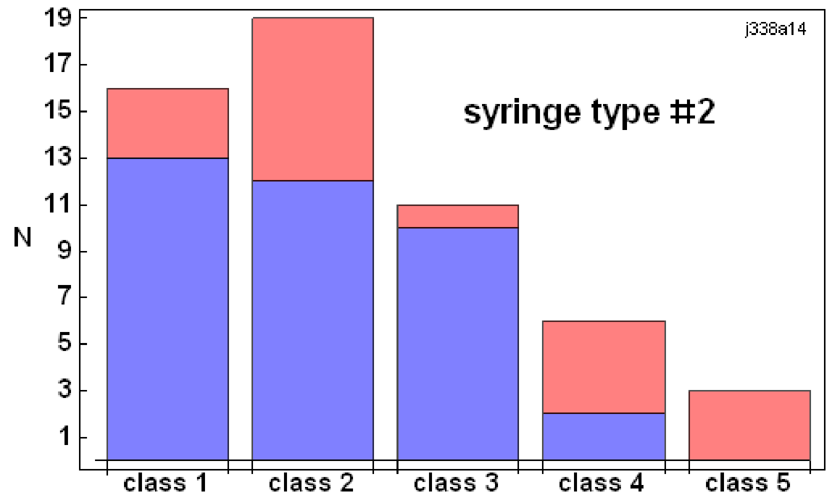


Visual appearance of flaws does not necessarily give a hint about the criticality

Size versus criticality of flaws (example)

- Two types (formats) of glass syringes
- Classification of flaws by (lateral) size
- Burst-pressure strength experiments
- Fractographic examinations (location of fracture origin)
    - Failure at classified defect?





Optical assessment does not yield a reliable information about flaw criticality

Definition of fractography

- ASTM C 1145: "Means and methods for characterizing a fractured specimen or component"
- Macroscopic fractography: Examination and interpretation of crack patterns
  - Failure-inducing mechanical tensile load
- Microscopic fractography: Examination of fracture-exposed surfaces and the interpretation of the fracture markings
  - Failure-inducing flaw
- Art or science to conclude the failure of brittle materials from fracture surfaces and patterns

Fractography enables an objective assessment of the circumstances of failure of a solid

Definition of fractography

Glass breakage: *surface flaw × tensile load ≥ breakage resistance*

Microscopic fractography

- Interpretation of fracture surface markings
  ↓
- Failure-inducing flaw

Macroscopic fractography

- Interpretation of crack patterns
  ↓
- Failure-inducing mechanical tensile load

Root cause for brittle failure

Fractography can answer many questions

- Position of failure-inducing flaw?
- Type of failure-inducing flaw?
- Direction of failure-inducing mechanical load?
- Type of failure-inducing mechanical load?
- Container closure-integrity affected?
- Velocity of failure propagation?
- (Magnitude of failure-inducing mechanical load?)
- (Static or dynamic failure?)
- (One-step or multiple step failure?)
- (Presence of corrosive medium?)

Initiation of failure (fracture)

- Application of mechanical load causes deformation (elastic strain)
- Elastic strain stores volume energy
- Impetus for failure: Release of stored volume energy
  - Release of energy by creation of surfaces ($\Rightarrow$ fracture surfaces)

Impetus for brittle failure: Release of stored elastic energy (creation of surfaces)

- Propagation perpendicular to (local) principle tension

Crack propagation direction *always* perpendicular to local principle tension

- Acceleration from v = 0 m/s up to maximum velocity ($\approx$km/s)
- Further release of energy by creation of additional surfaces $\Rightarrow$ branching

Crack branching starts at maximum propagation velocity

Fracture patterns (macroscopic fractography)

- Shape/orientation of cracks gives hints about direction of mechanical load
- Deduction of load situation
  - Constant or inhomogeneous
  - Bending
  - Side compression
  - Thermal gradients
  - Inner pressure
- Branching
  - Backtracking to first branching ⇒ vicinity of fracture origin
  - Maximum crack propagation velocity reached

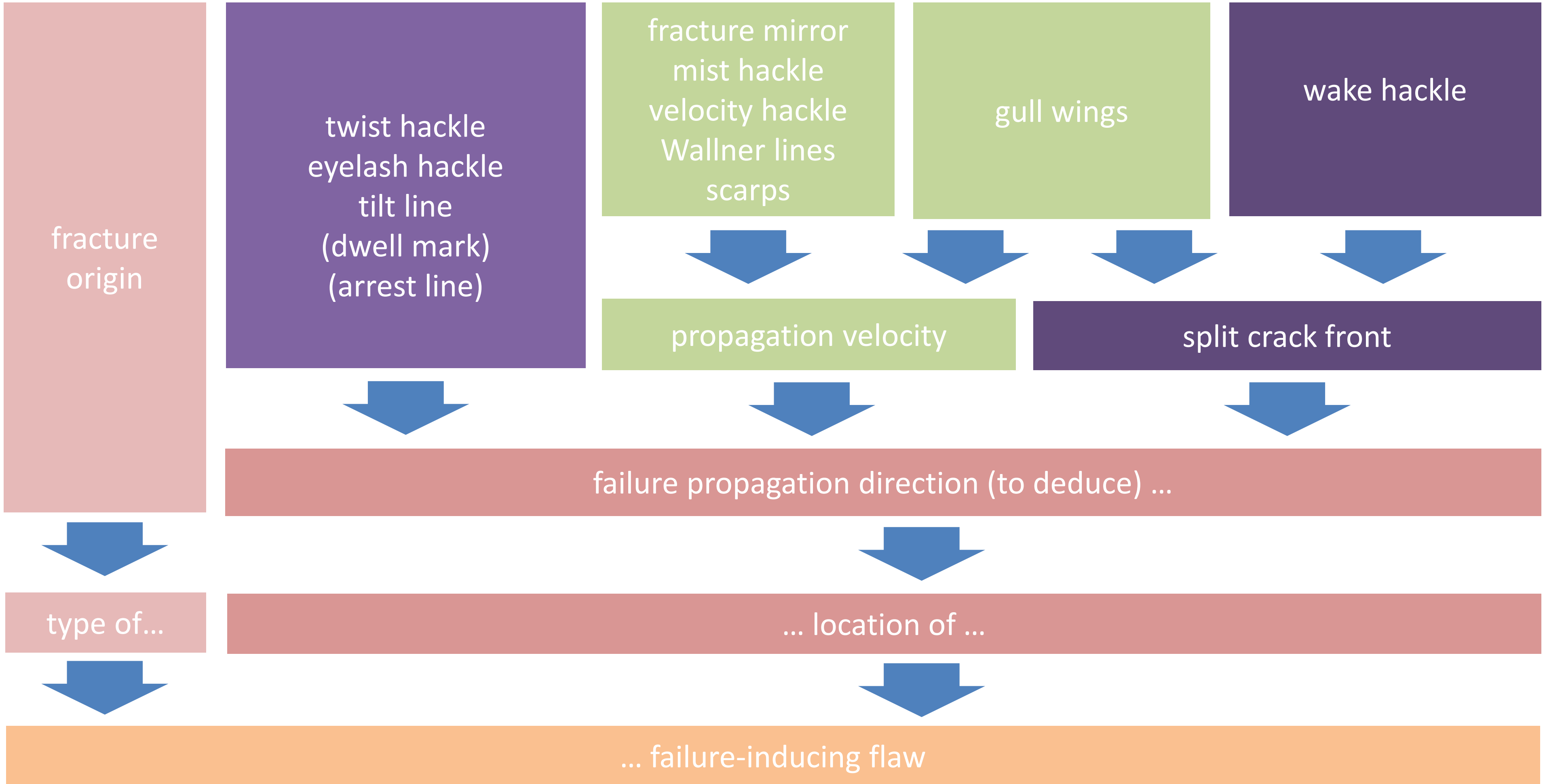
Macroscopic fractography is capable to characterize the failure-inducing mechanical load

Fracture surface markings (microscopic fractography)

- Topographic features generated during crack propagation
  - Fracture mirror
  - Mist/velocity/twist/wake/eyelash hackle
  - Wallner lines, gull wings
  - Tilt/arrest line, dwell mark
  - Chipping
  - Scarps
- Observation gives hints about propagation conditions
  - Failure propagation velocity
  - Failure propagation direction
  - Change of direction and/or magnitude of mechanical load
  - Split crack front
  - …

Microscopic fractography is capable to determine the fracture origin position

Information content of fracture characteristic surface markings



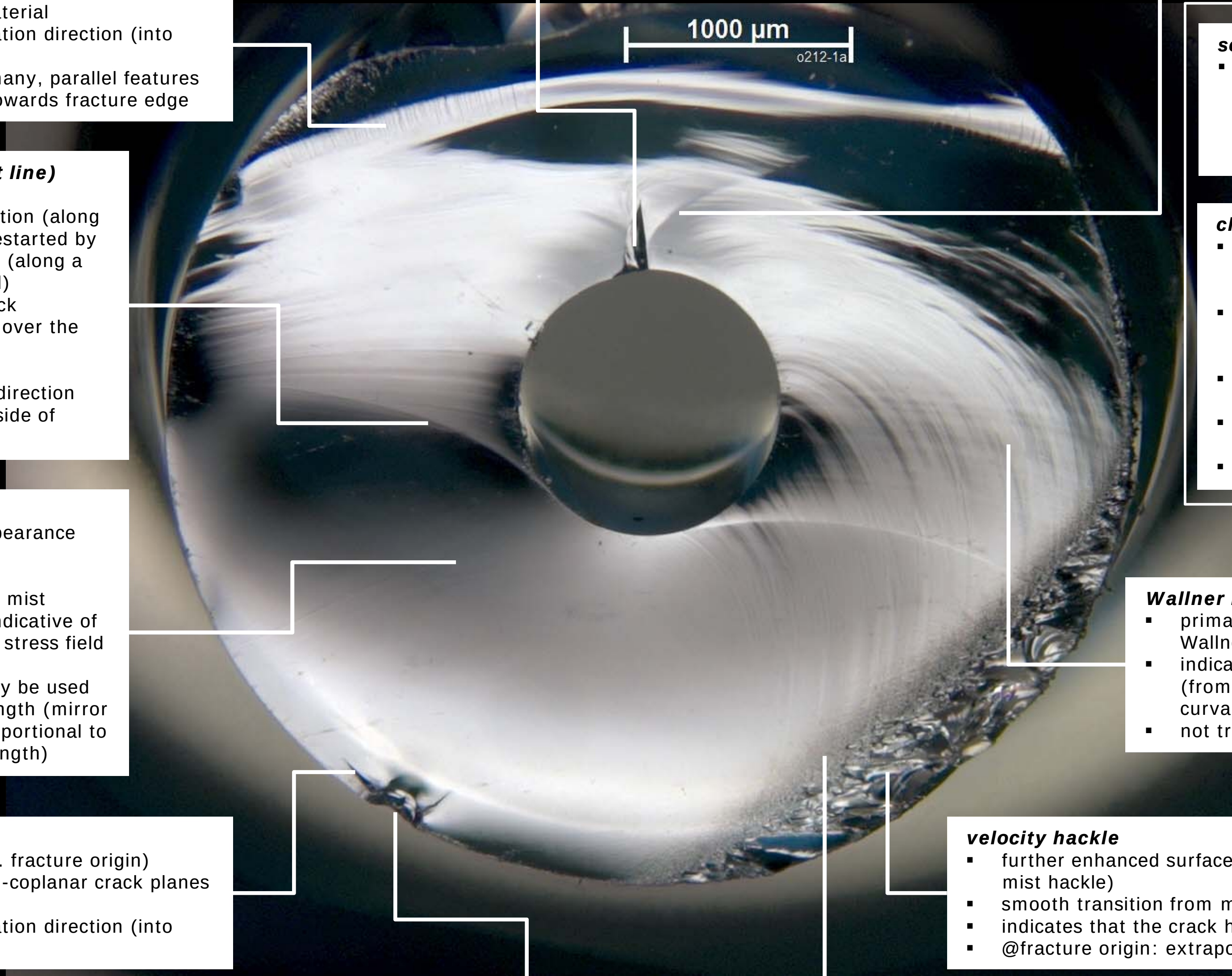
Fractography Cheat Sheet

wake hackle
- formation requires reunion of two split, non-coplanar crack fronts after surrounding a discontinuity
- occurrence as only one hackle
- indication of local propagation direction (into direction of hackle taper)

gull wings
- special case of (primary) Wallner line
- formation requires reunion of two split crack fronts
- indication of propagation direction (from concave to convex side of curvature)
- not true shape of crack front

twist hackle
- occurrence within bulk material
- indication of local propagation direction (into direction of hackle taper)
- often appear as sets of many, parallel features
- often pointing outwards towards fracture edge

tilt line (dwell mark / arrest line)
- indication for either
  - stop of crack propagation (along a given plane) and restarted by a different stress field (along a new plane or changed)
  - sudden change of crack propagation direction over the whole crack front
- true shape of crack front
- indication of propagation direction (from concave to convex side of curvature)



scarps (not shown)
- formation requires corrosive medium at crack front (water, acid, base) and slow crack propagation velocity

chipping (not shown)
- formed by shear-stresses (surface near/flat-angled mechanical load)
- in company with tertiary Wallner lines hint for dynamic contact
- often dwell marks on surface
- flat, conchoidal, flake-like fragments
- often as secondary failure

(fracture) mirror
- smooth surface, shiny appearance
- area of low crack velocity
- at fracture origin:
  - shape (defined by the mist hackle boundary) is indicative of the distribution of the stress field at the time of failure
  - mirror dimensions may be used to determine the strength (mirror radius is inversely proportional to the square of the strength)

Wallner lines
- primary, secondary, tertiary Wallner lines
- indication of propagation direction (from concave to convex side of curvature)
- not true shape of crack front

eyelash hackle
- starting from surface (e.g. fracture origin)
- formed by merging of non-coplanar crack planes at the surface
- indication of local propagation direction (into direction of hackle taper)

velocity hackle
- further enhanced surface roughness (compared to mist hackle)
- smooth transition from mist hackle to velocity hackle
- indicates that the crack has reached terminal velocity
- @fracture origin: extrapolation possible

fracture origin
- location from where failure initiated (highest stress intensity was reached first)
- may contain the stress concentrator or it may be the stress concentrator.

mist hackle
- region with enhanced surface roughness
- indicates that the crack has nearly reached terminal velocity
- may define the boundary of the mirror region

SCHOTT
glass made of ideas

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October 1, 2017

Strength and fractography of glass

- The strength of glass is not a material constant
- The strength of glass is a projection of the surface quality
    - Defined by flaw type and size distribution(s)
    - The strength of glass can be described by statistical distributions
- Generation of new flaws during processing can reduce the overall strength
- Critical flaws (in terms of strength) cannot be compared to cosmetic flaws
    - Optical inspection techniques are not sensitive for critical flaws
    - Risk of wrong release criteria

Further reading

- Haines, D. et al.: “Why do Pharmaceutical Glass Containers Break: The Underestimated Power of Strength Testing and Fractography”; International Pharmaceutical Industry 8/1 (2016) 88.
- Haines, D. et al.: “Die Anwendung von Festigkeitsprüfungen und Fraktografie auf pharmazeutische Glasbehälter”; Pharm. Ind. 78/8 (2016) 1208.
- Quinn, G.D.: “Fractography of Ceramics and Glasses”; NIST Special Publication 960-16e2 (2016).
- Parenteral Drug Association: “Technical Report No. 43: Identification and Classification of Nonconformities in Molded and Tubular Glass Containers for Pharmaceutical Manufacturing: Covering Ampoules, Bottles, Cartridges, Syringes and Vials”; revised 2013

Thank you for your attention!