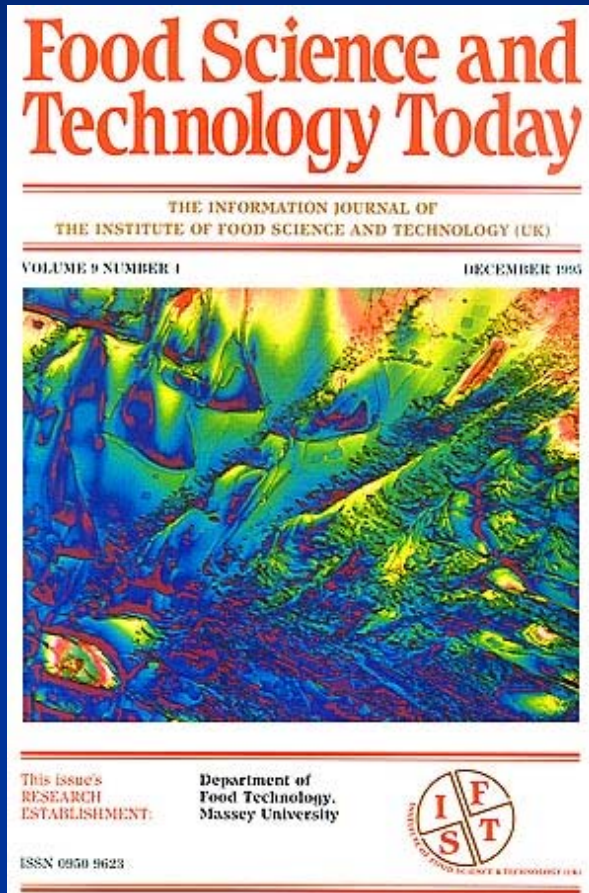


Use of synchrotron radiation microscopy in food research



Franco Zanini
Sincrotrone Trieste

Food science and microscopy



- Why microscopy is used to study the quality of foods?
- Why the microstructure of food is so important?

Food science and microscopy

- Food technology is a controlled attempt to preserve, transform, create or destroy a structure imparted by nature or processing
- Microscopy and imaging techniques available to probe into the structure of food and rationally design processes that enhance the quality of products
- Image analysis and image processing provide the quantitative data for the analysis and design of food microstructure

Food science and microscopy

Food microstructure and interaction of components (proteins, fat, carbohydrates, water, etc.) is related to:

- Nutrition
- Chemical stability
- Microbiological stability
- Texture and physical properties
- Transport properties
- Product engineering

Structure of bread crumb



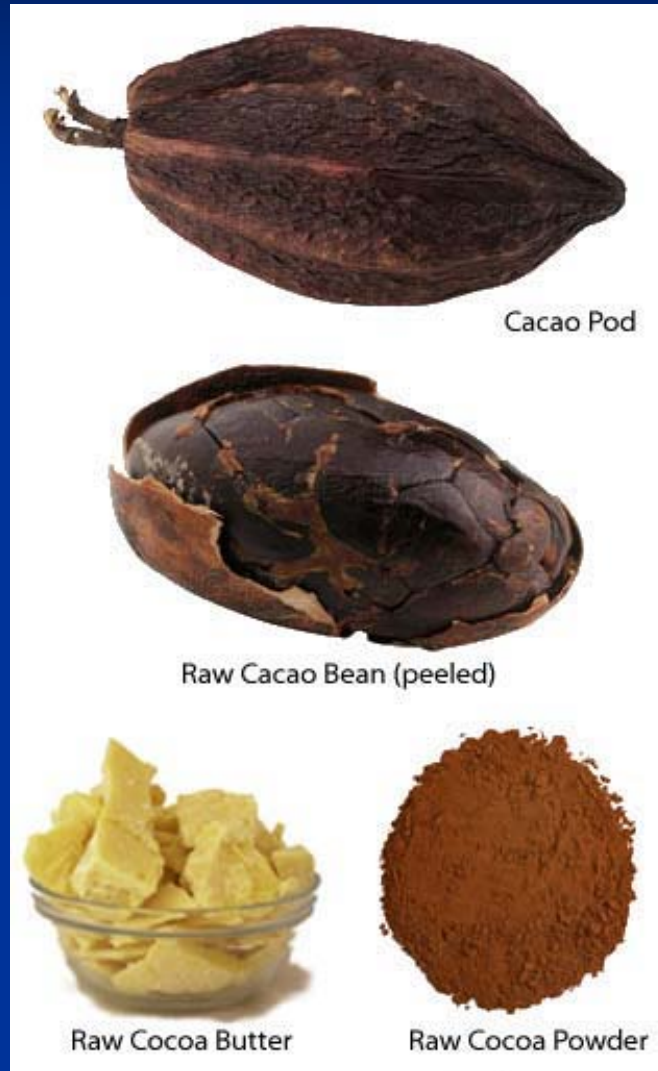
- Cell wall thickness, cell size and uniformity of cell size affect the texture of bread crumb
- Appearance, taste perception, stability of final product

Structure of dairy products



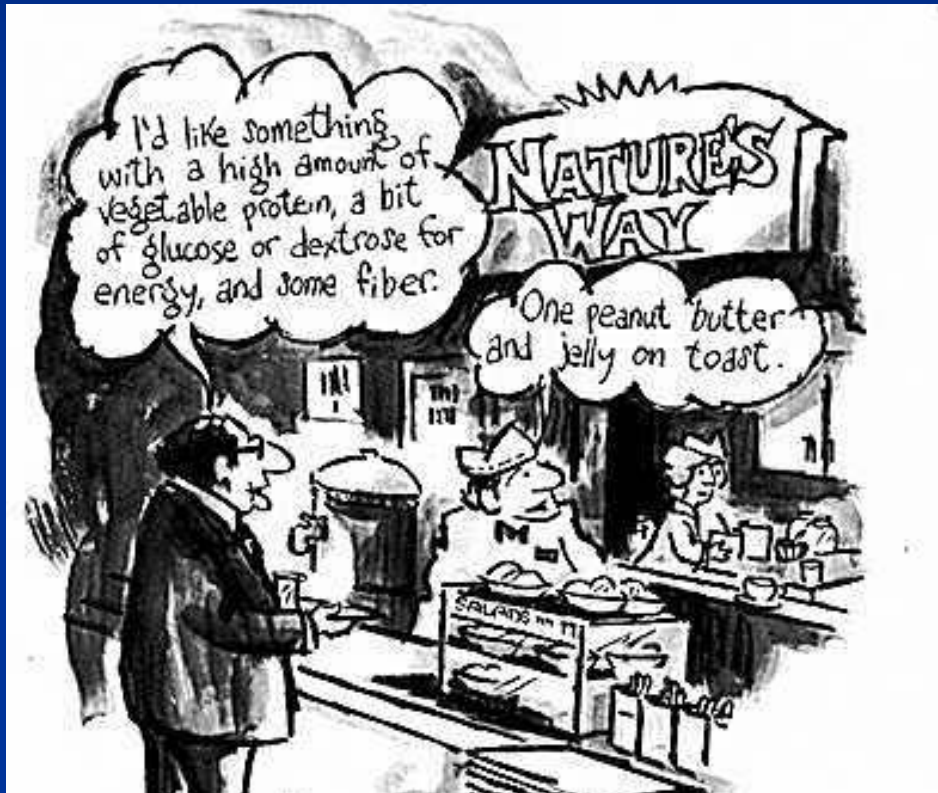
- The structural organization of cheese (protein network) affects the cheese texture
- Regular and close protein matrix, small and uniform fat globules -> more elastic behavior

Structure of cocoa butter



- Polymorphic transformation of fat crystals and coexistence of different polymorphic forms affect the mechanical properties of cocoa butter
- Fast-heated chocolate samples have pores smaller than the slow-heated ones, and require a higher energy to determine a fracture in the final product

Structure of peanut butter



- A study carried out on the effects of grind size on peanut butter texture demonstrated that an increase of that variable decrease spreadability, sensory smoothness and adhesiveness

Techniques for microstructure studies

Optical Microscopy

- Requires a minimum sample preparation
- Allows specific staining
- Modest magnification

Scanning and Transmission Electron Microscopy

- High spatial resolution
- Allows elemental analysis
- Complex sample preparation leading to artifacts

Techniques for microstructure studies

Confocal Laser Scanning Microscopy

- Requires a minimum sample preparation
- Real 3D information

Magnetic Resonance Imaging

- Non-destructive technique
- Suitable for dynamic studies

Atomic Force Microscopy

- Non-destructive technique
- High spatial resolution

X-ray imaging at a 3rd generation SR facility

- high energy photons and high flux
- heavy and/or bulky samples in transmission geometry
- tunability in a large energy range
- short exposure times
- small angular source size and big source-to-sample distance
- use of natural coherence properties of the beam

The SYRMEP beamline:

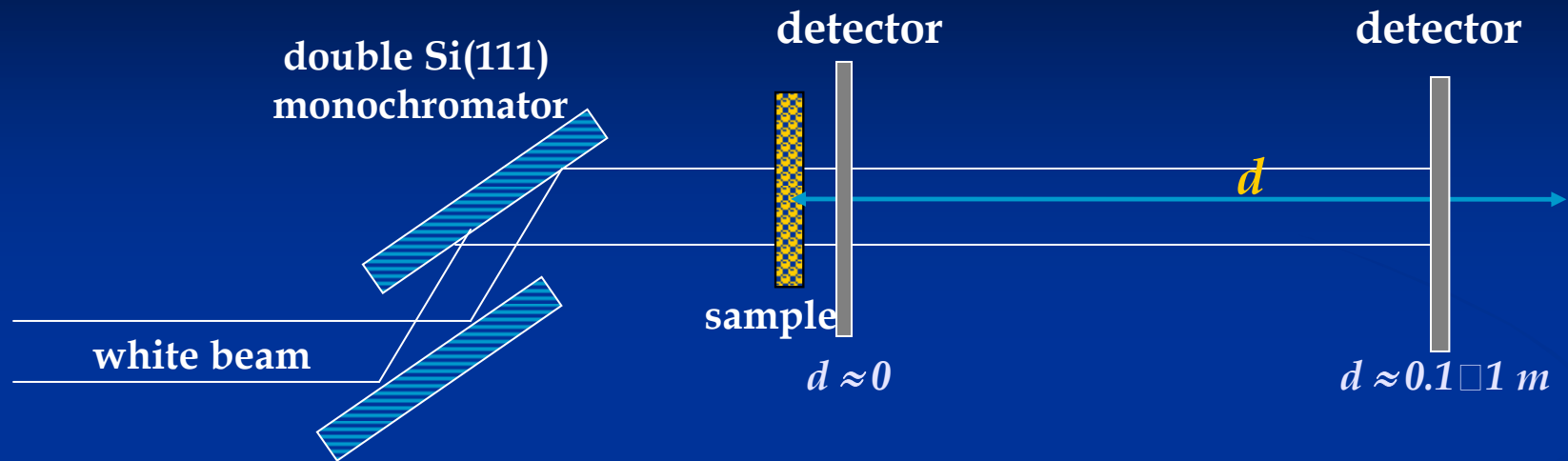
Source size σ (h x v) = 1100 μm x 100 μm

Source-to-sample distance: D = 24 m

Beam size at sample (h x v) = 150 mm x 6 mm

Energy range: 8 ÷ 35 keV, Bandwidth: $\Delta\lambda/\lambda = 2 \times 10^{-3}$

Absorption and Phase Sensitive (PS) Radiography



$$(\Delta I/I)_{\text{abs}} = e^{-c \Delta \mu} - 1$$

$$\Delta \phi = 2\pi c \Delta \delta / \lambda$$

$n = 1 - \delta - i\beta$: refraction index

$m = 4\pi \beta / \lambda$: linear absorption coeff.

c : object size // to beam direction

$r \ll a \Rightarrow$ edge detection regime

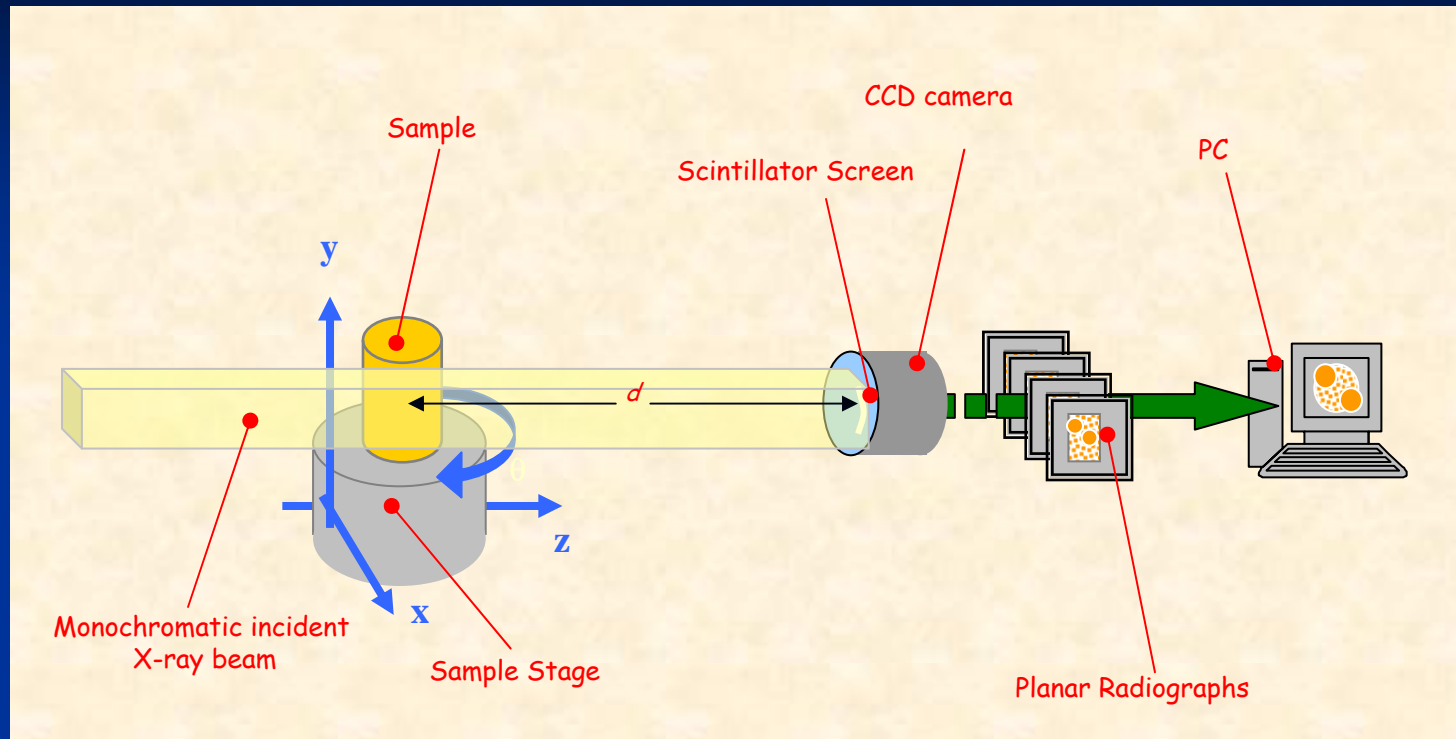
$r \approx a \Rightarrow$ holographic regime

$r \gg a \Rightarrow$ Fraunhofer diffraction

a : object size \perp to beam direction

$r = (\lambda d)^{1/2}$: first Fresnel zone radius

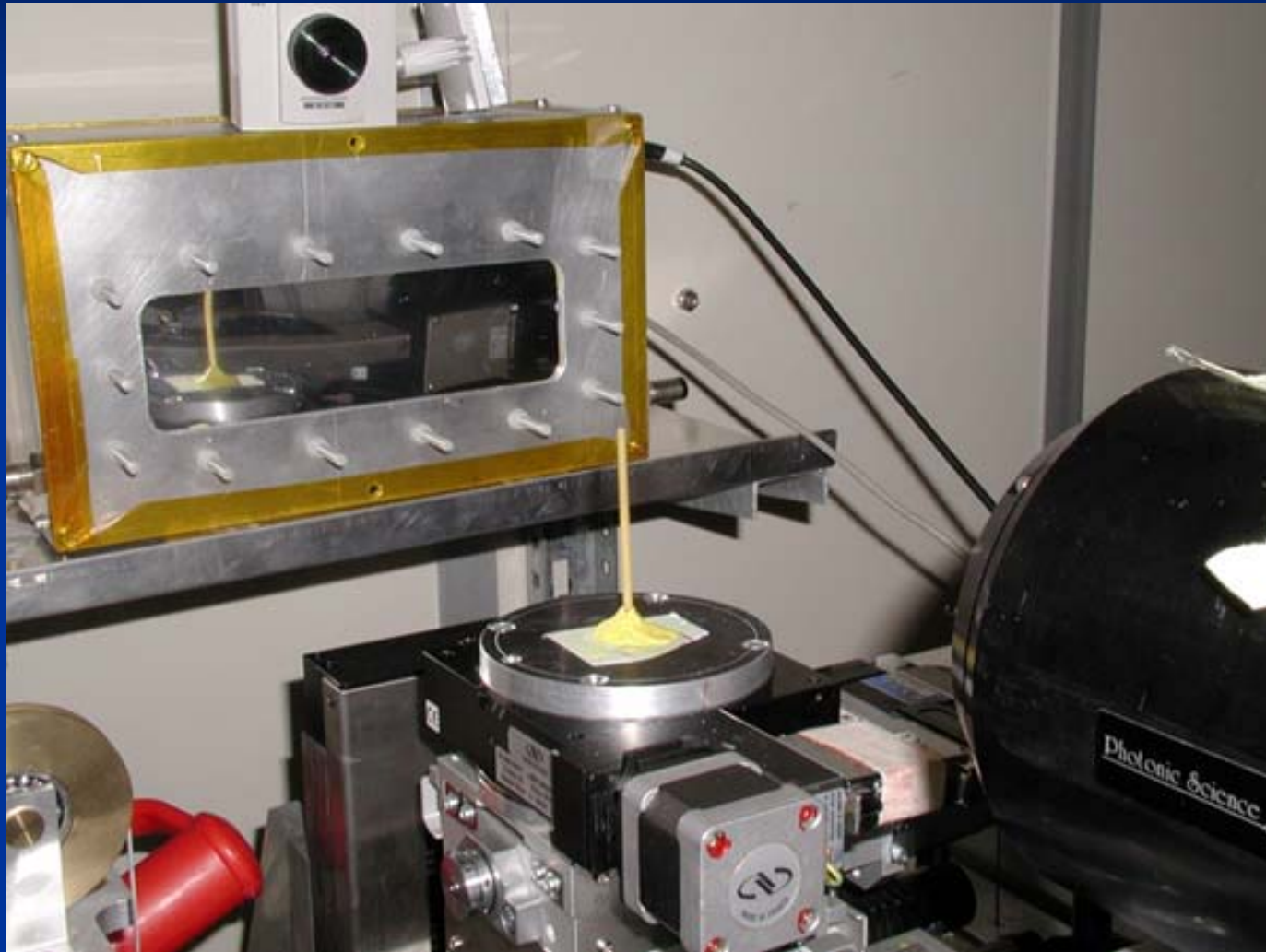
Absorption and PS Computed μ -Tomography (μ -CT)



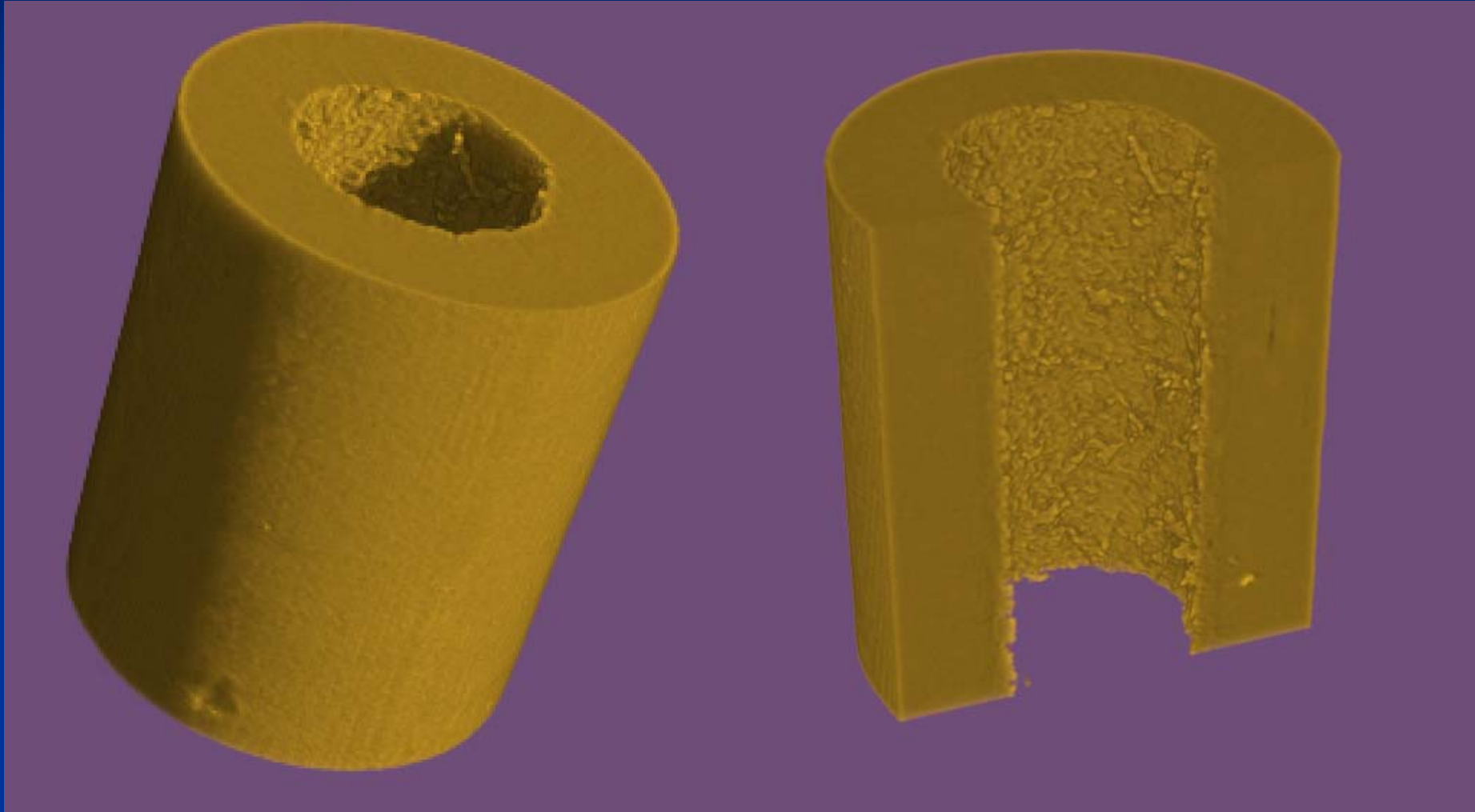
CCD camera: 4008×2672 pixels², pixel size: $4.5 \mu\text{m}$, FOV: $18 \times 12 \text{ mm}^2$

- Precious for investigation of internal features without sample sectioning:
- in many cases the sectioning procedure modifies the structure of the sample
- the sample can be after studied by other experimental techniques,
- or submitted to several treatments (mechanical, thermal, etc...)

Microtomography of industrial pasta



Microtomography of industrial pasta



Quantitative analysis of bread crumb

- Phase sensitive micro-tomography as a non destructive imaging technique for the reconstruction of bread crumb microstructure
- Stereological analysis performed to determine void volume fraction, cell wall thickness, favorite orientation of cell structure and degree of anisotropy

QuickTime™ e un decompressore sono necessari per visualizzare quest'immagine.

Quantitative analysis of bread crumb

QuickTime™ e un
decompressore
sono necessari per visualizzare quest'immagine.

Stress-strain curves for observed and
calculated mechanical behaviour of different
bread samples

P.M. Falcone et al., J. Food Sci. 70, 266
(2005)

- By comparing results from mechanical tests and those from image analysis it is possible to observe that bread elastic properties are closely related to its structural parameters.
- The sample with highest elastic modulus has also the lowest porosity, the highest value of cell wall number and specific surface of solid phase.

Quantitative analysis of bread crumb

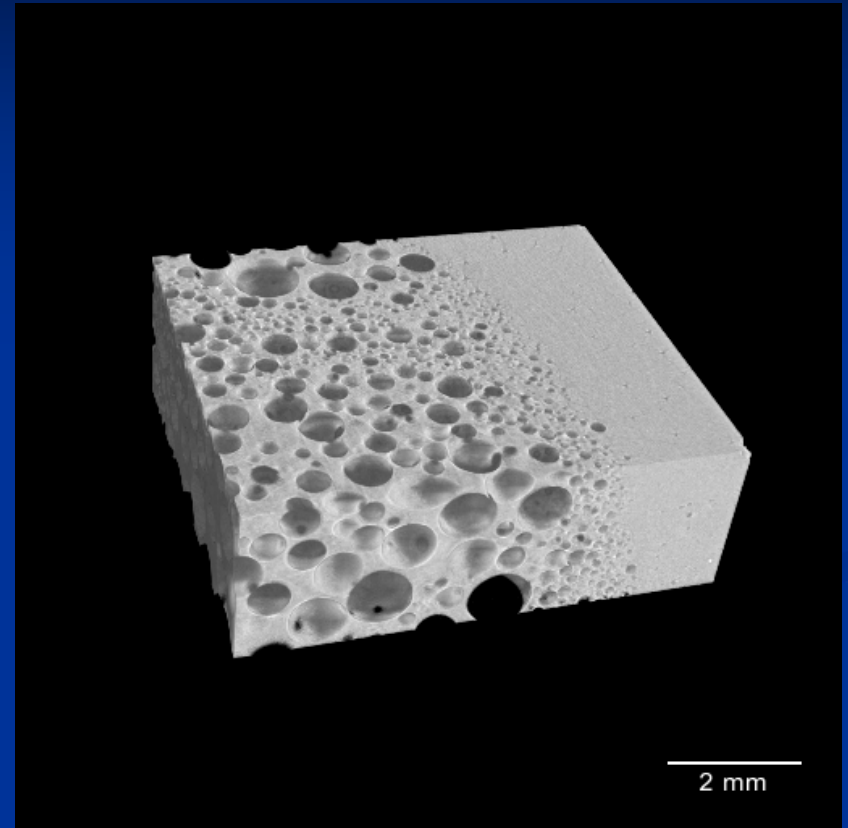
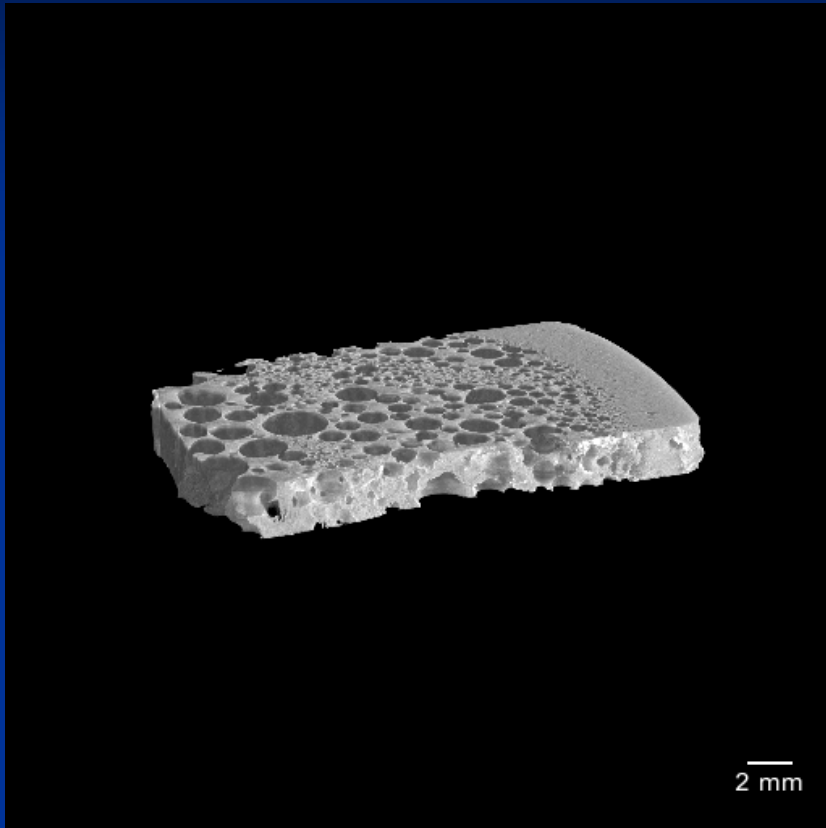
QuickTime™ e un
decompressore
sono necessari per visualizzare quest'immagine.

Trend in the anisotropy of the volumes of
interest (VOI) within two different samples.

P.M. Falcone et al., J. Food Sci. 70, 266
(2005)

- Evaluation of the anisotropy degree within different samples shows a preferential and different orientation of the solid phase.
- 3D analysis is the only approach which can give directionally dependent parameters in order to describe bread crumb and, in general, foamed food structures.

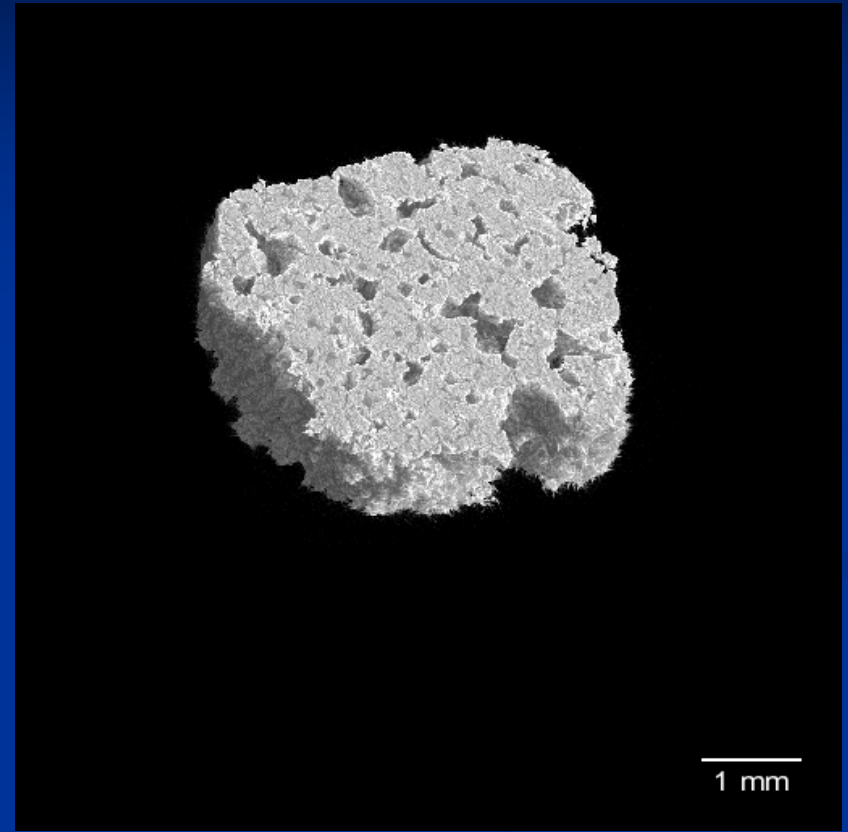
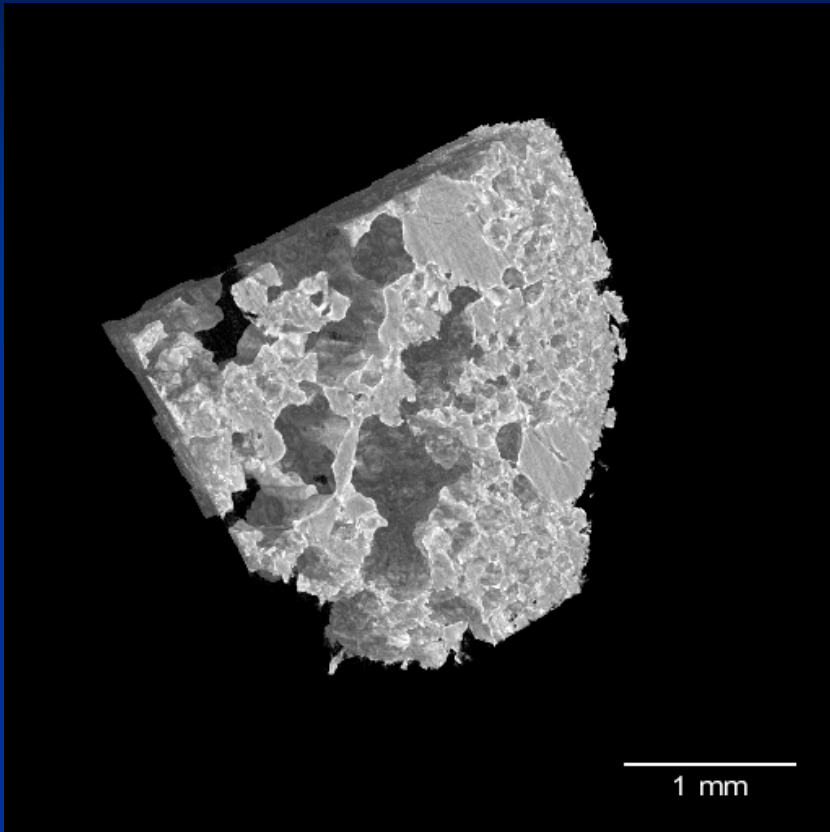
3D structural analysis of foamed food



Volume rendering of virtual sections (100 and 200 slices) of milk aerated chocolate bar revealing the foamed structure of the sample.

P.M. Falcone et al., in *Advances in Food and Nutrition Research*, vol. 51, p. 205 (2006)

3D structural analysis of sponge-like food



Volume rendering of commercial and home made crust pastry biscuits showing a highly interconnected structure. Different textures imply different sensorial characteristics.

P.M. Falcone et al., in *Advances in Food and Nutrition Research*, vol. 51, p. 205 (2006)

Textural properties of coffee beans



- The brittleness and fragility induced by roasting is the primary attribute of roasted coffee beans.
- The investigation of water content and state of structural properties of coffee beans is of great interest for process purposes to limit textural changes during shelf-life.

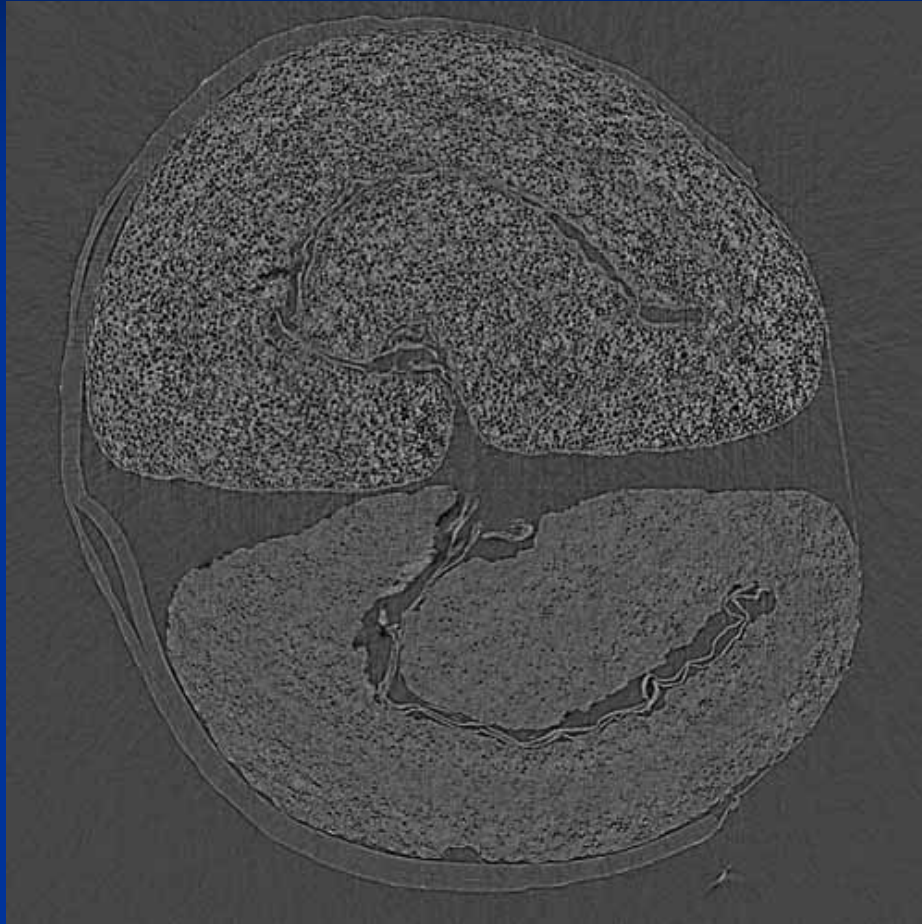
Textural properties of coffee beans

- Roasted beans present a **lower strength and toughness** than raw ones and, independently from roasting, resulting textural properties are **highly dependent on water activity**.
- At increasing hydration degree, an **increase in strength and toughness** are observed until a critical value is reached.
- Above this value **plasticization** occurs.

QuickTime™ e un
decompressore
sono necessari per visualizzare quest'immagine.

P. Pittia et al, J. Texture Studies 38, 116
(2007)

Textural properties of coffee beans



- X-ray microtomography provides a **digital description** of the coffee bean texture as a function of **moisture and water activity**.
- These values are of great interest in relation to the grinding step carried out on roasted coffee beans before the extraction of coffee brew.

Internal slice of two *Coffea Arabica* beans
(field of view = $4 \times 4 \text{ mm}^2$, pixel size = $4.5 \text{ }\mu\text{m}^2$)

Vibrational microspectroscopy

- Raman microspectroscopy offers selective image contrast of each molecular component with spatial resolution of about $1\ \mu\text{m} \times 1\ \mu\text{m}$
- Infrared microspectroscopy (IR) can provide complementary information (water is invisible in Raman) with spatial resolution of about $20\ \mu\text{m} \times 20\ \mu\text{m}$
- Synchrotron IR uses a source 1000 times brighter, highly directional and without thermal noise

Granular structure of resistant starches

QuickTime™ e un
decompressore
sono necessari per visualizzare quest'immagine.

SEMs of normal
corn starch citrate

QuickTime™ e un
decompressore
sono necessari per visualizzare quest'immagine.

Xie et al., Food Res.
Int. 39, 332 (2006)

- Resistant starch (RS) in the diet is able to lower the glycemic response in humans and to reduce the incidence of type II diabetes
- RS can also be used to improve cholesterol metabolism and reduce the risk of colon cancer

Granular structure of resistant starches

QuickTime™ e un decompressore sono necessari per visualizzare quest'immagine.

- Citric acid treatment induces changes in the morphology of starch granules
- A new peak at 1724 cm^{-1} (ester bond) was observed in Fourier Transform IR spectroscopy, indicating starch citrates are heat-stable.

SEMs of high amylose corn starch citrate

QuickTime™ e un decompressore sono necessari per visualizzare quest'immagine.

Xie et al., Food Res. Int. 39, 332 (2006)

Distribution of ions and molecules in developing and mature wheat grain

QuickTime™ e un decompressore sono necessari per visualizzare quest'immagine.

- Understanding subtle differences in the composition of cereal grain is important for the optimisation of its utilisation
- An important goal is to link compositional information with material properties such as biomechanical properties

FT-IR microscopy of transverse sections of grain of wheat grown under different conditions

Mills et al., J. Cereal Sci. 41, 193 (2005)

Acknowledgments

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