Politechnika Wrocławska

Specimen Preparation for SEM investigation

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Presentation program

- Aim of SEM investigation
- Investigated materials
- Condition for specimens
- Preparation
- Specimen fixation
- Replica
- Examples

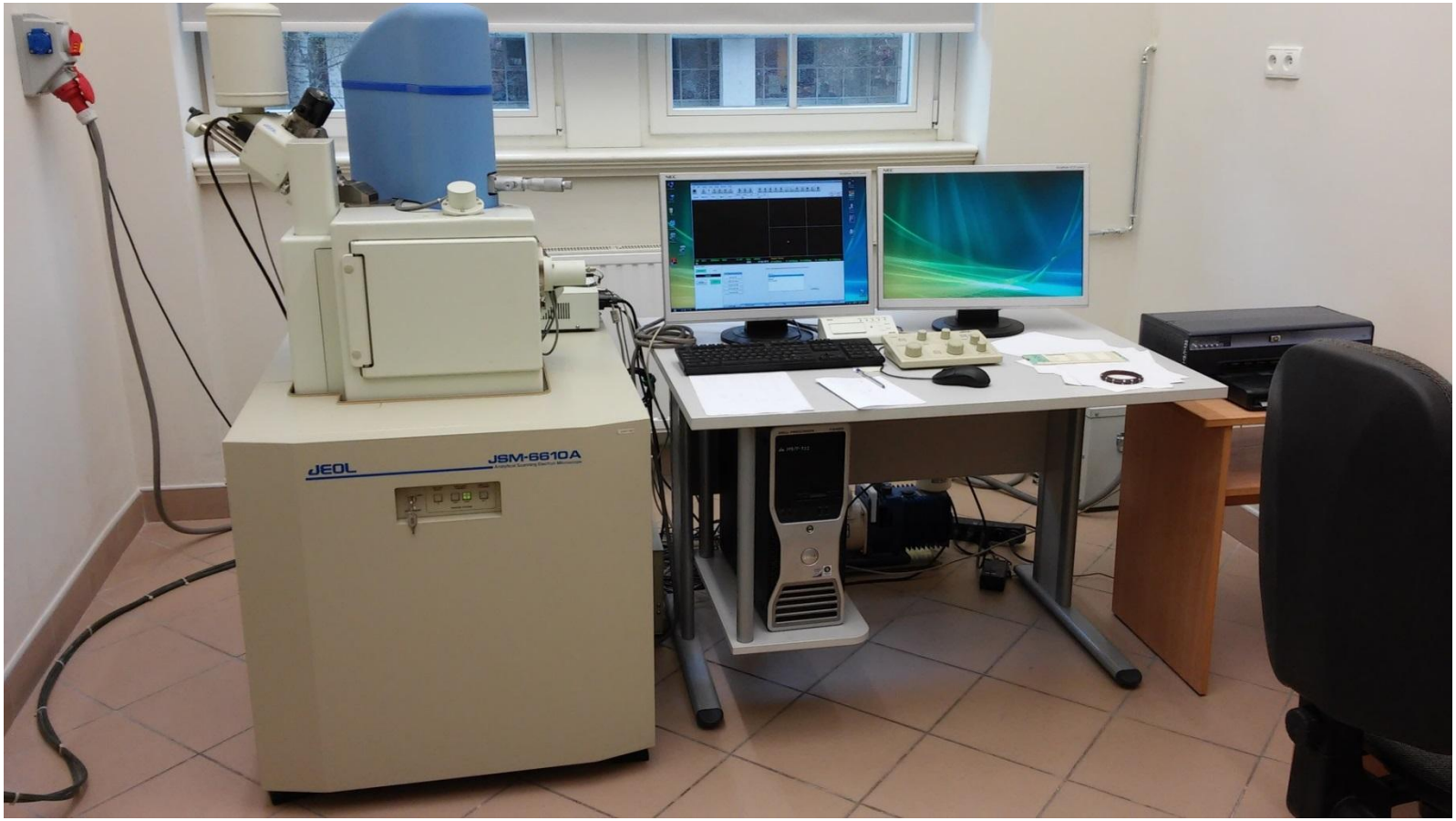
Aim of SEM investigation

Materials are investigated for:

- Mikrostructure determination (SE, BSE, AE, EBSD – Electron Beam Selected Diffraction)

- Chemical composition: (EDS, BSE,AE)

Analytical SEM JEOL JSM-6610A

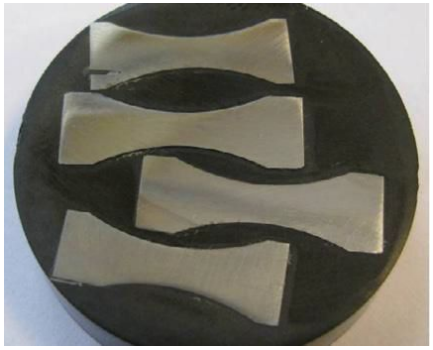


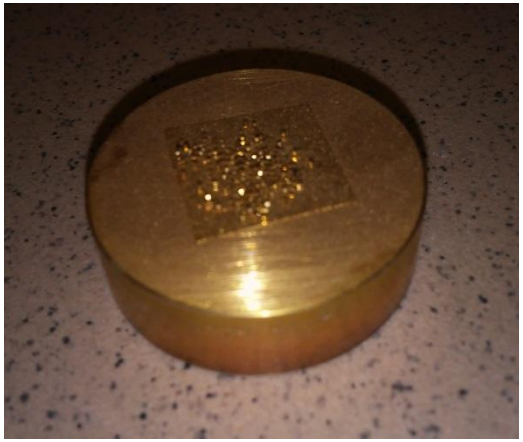
Low vacuum SEM JSM- 5800LV

Types of specimens for SEM investigation

Four types of specimens:

1. Metalic
2. Polymer
3. Biological
4. Geological





Metalic specimens

For current conductive metalic specimens any additional preparation is not necessary.

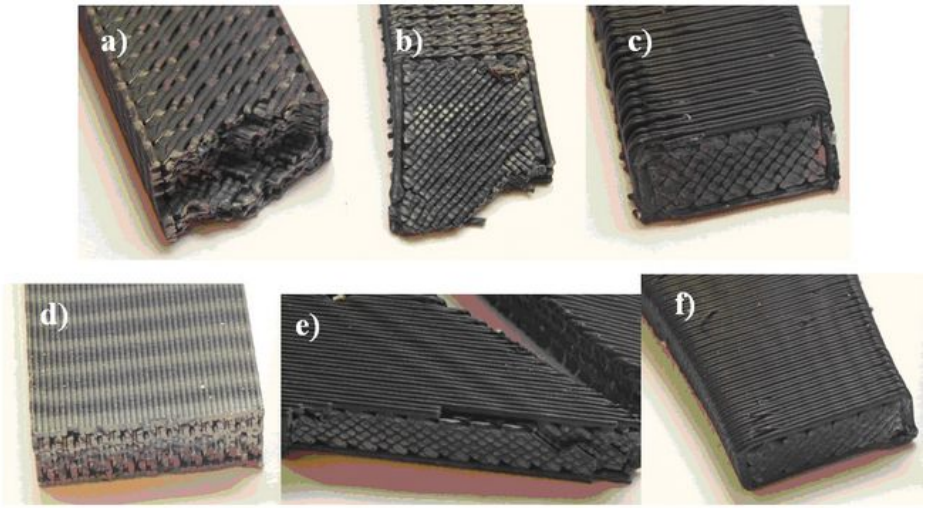
They can be investigated like specimens for macro or metallography research.

Specimen can be at polished or tobe at etched state.

It is only necessary to fix the specimen with appropriate holder.

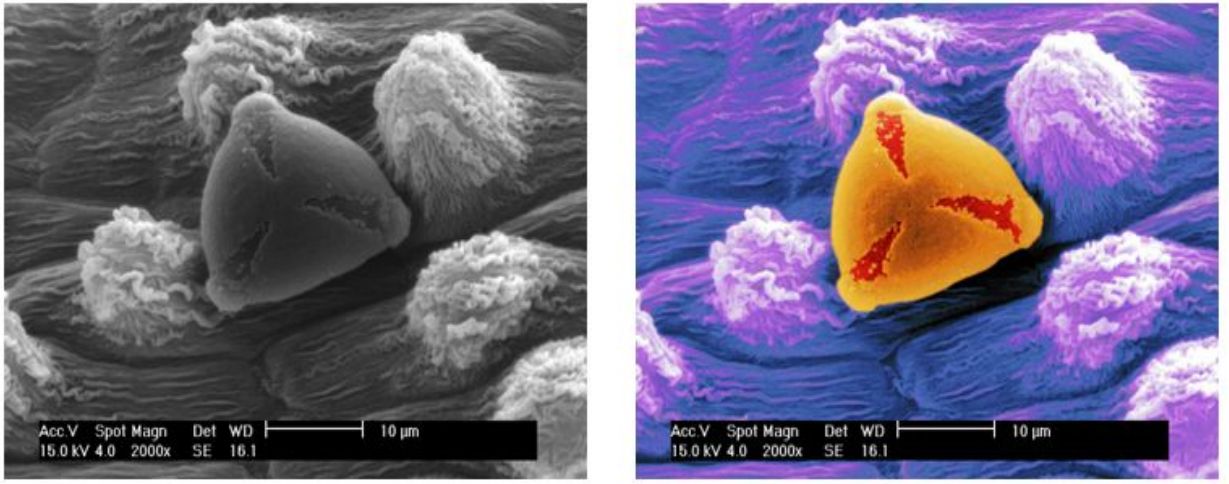
Specimens from polymers and composites

Polymer specimens must be sputtered by the layer of current conductive elements like: C, Au, Pt, Cu.



Biological specimens

Living cells, biological tissue, and some organs needs to be specially prepared for the reason their fixation and protection to stabilize them and to protect against the ravages of the electron beam.



SEM image of pollen before and after graphic processing

Biological specimens

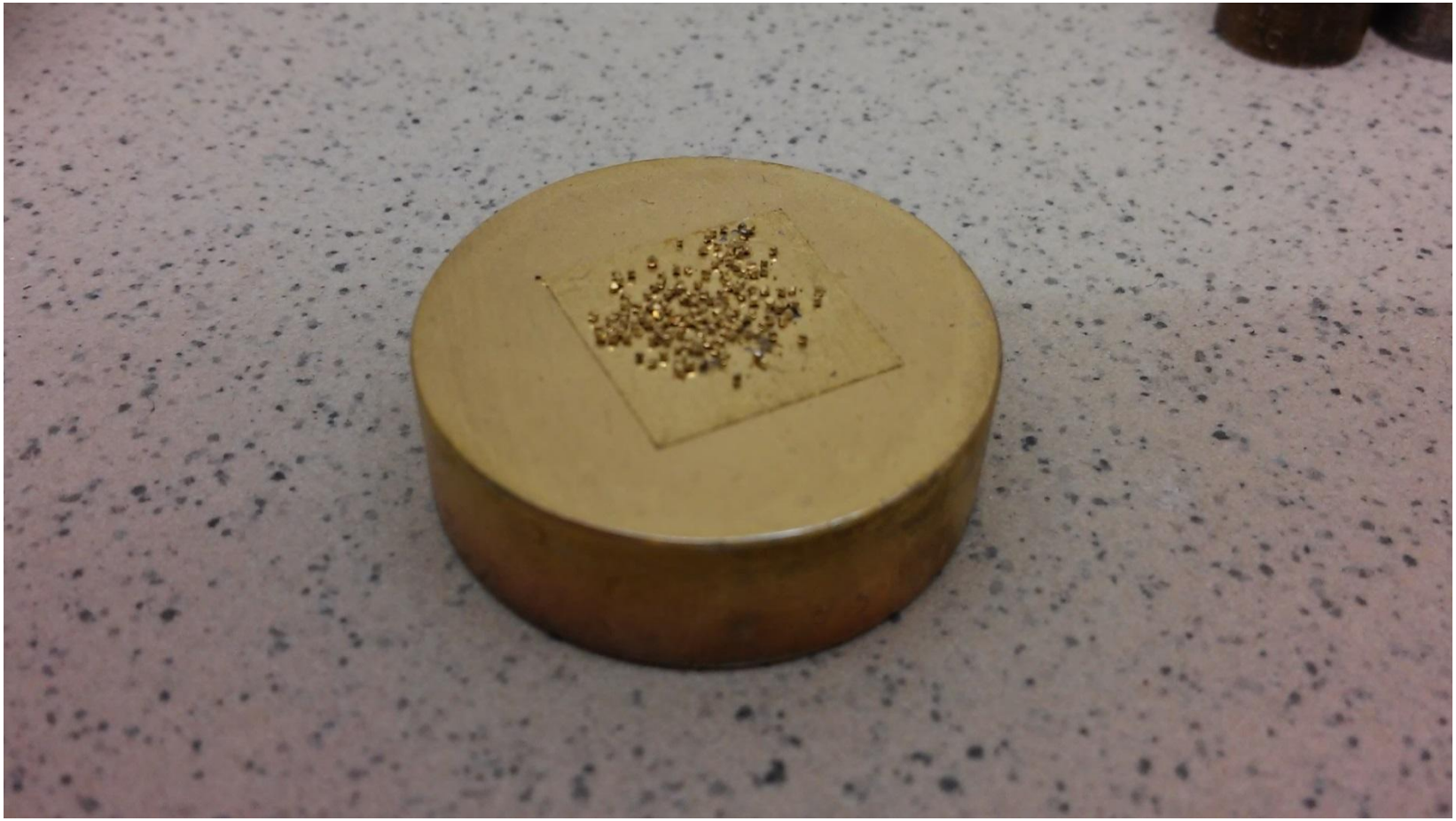
Biological specimens must be:

1. Dried, because inside the SEM chamber the material will be in the vacuum and therefore can not be inserted preparations hydrated.

2. Sputered by current conductive material. Carbon is the best.

Flower petals sprinkled by gold

Samples of powder sputerred by gold



Biological specimens covered by gold

Specimen size

Specime sizes are limited by dimensions of SEM support table. Typical values are:

- Diameter below 5cm,
- Highest below 2,5cm.

Typical dimensions are: 10 x 10 x 5 mm.

Specimens embeding

Specimens are embeded at epoxy resin for the reason of better mounting and correction of specimen quality.
Before mounting specimens must be cleaned, free of dust, grease and any impurities.

Two techniques can be applied:

- Hot embeding under the pressure,
- Cold embeding.

Specimen embeding

- „Cold embeding” is suitable for materials sensitive at high temperature and pressure. Special epoxy or acryl resines are applied.
- „Hot embeding” is suitable in the case when high quality of specimen preparation, equal size, shape and short time preparation is necessary. This process is realized by special equipment, (hot temperature press pressure).

Electrical current conductivity

Specimens analyzed by SEM methods must conduct electrical current.

If specimen doesn’t conduct electrical current, then must be covered by the layer of Au, Pt, C or Cu.

Such prepared specimens can be investigated at high or low vacuum.

Specimen preparation

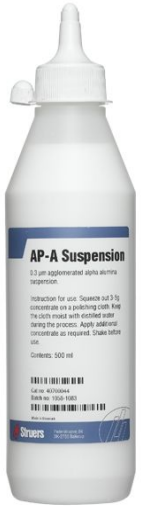
- Cutting, to obtain dimensions limited by support table disposed inside specimen chamber
- Cleaning and degreasing of specimen surfaces
- Grinding
- Polishing
- Etching

Specimen preparation

Grinding, by using special waterproof fine grain grinding papers.

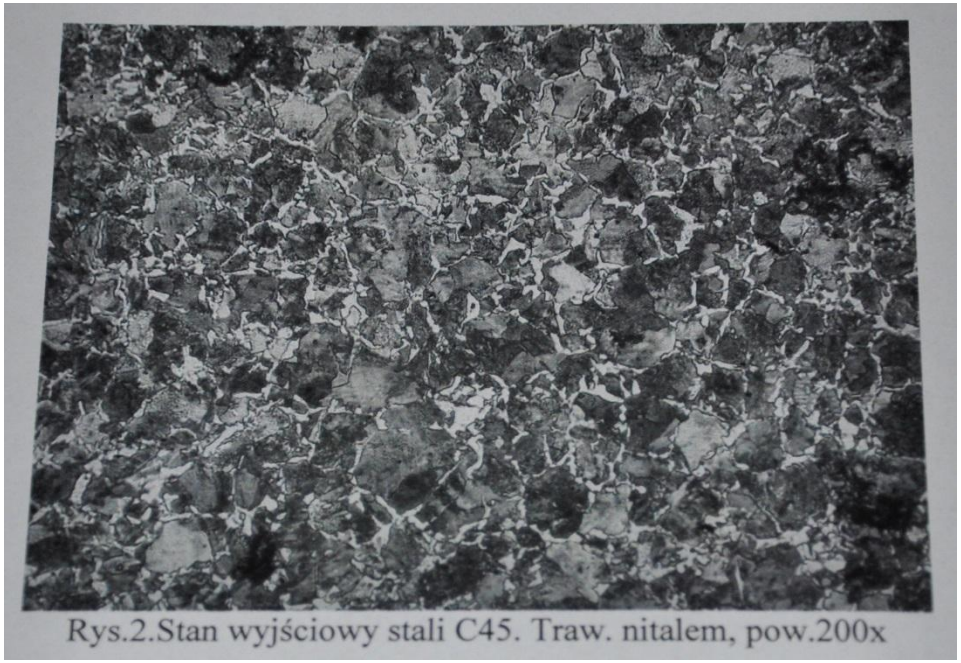
Specimen preparation

Mechanical polishing by using special velvet tissue immersed by diamant paste or water suspension of Al2O3. Any traces of scratches must be eliminated. Specimen surface must be brillant.



Specimen preparation

Etching is the last operation necessary for microstructure visualisation. It is realised by application chemical reagent on the surface of polished specimen.



Rys.2.Stan wyjściowy stali C45. Traw. nitalem, pow.200x

Etching reagents

Rodzaj stopu	Skład odczynnika	Sposób trawienia	Zastosowanie
Stopy Fe-C	Nital - MilFe 1 - 5cm^3 HNO_3 + 100 cm alkoholu etylowego	Kilka sekund do kilku minut w zależności od rodzaju i stopu	Ujawnia granice ziaren i składniki strukturalne stopów
Stopy Fe-C	Pikral - Mi3Fe 2—5 cm^3 kwasu pikrynowego +100 cm^3 alkoholu etylowego	Kilka sekund do kilku minut w zależności od rodzaju stopu	jw.
Stale węglowe i niskostopowe	Zasadowy pikrynian sodu — Mi9Fe 25 g NaOH + 2 g kwasu pikrynowego + 75 g H_2O	Temp. 60~100°C, kilka do kilkunastu minut	Zabarwia na brunatny kolor cementyt i azotki żelaza - ferryt pozostawia jasny
Stale węglowe i stopowe do ulepszenia	Mi7Fe 5 cm^3 kwasu pikrynowego + 0,5% alkilosulfonianu sodu (teepol) +100 cm^3 H_2O	Temp. ok. 55°C, czas traw. 1 - 3 min	Ujawnia granice ziaren austenitu w stalach zahartowanych i odpuszczonych
Stale stopowe chromowe i szybkotnące	Mi13Fe 10 g żelazicyjanku potasu + + 10 g wodorotlenku potasu + + 100 ml H_2O	W temp. pokojowej	Do stali stopowych narzędziowych. Trawi węgliki stopowe. Do stali 18-8. Trawi wysokotemperaturowy ferryt

Etching reagents

Stopy miedzi	Mi23Cu 100 g nadsiarczanu amonowego +1 dm3 wody	Trawienie na zimno lub w 60°C, płukanie wodą lub alkoholem	Ujawnia granice ziaren
Stopy aluminium	Mi1Al 0,5 cm3 HF (1,13) ++ 99,5 cm3 H20	Temp. 20°C, płukanie wodą lub alkoholem	Ujawnia granice ziaren i fazy międzymetaliczne
Stopy aluminium	Mi7Al 1 g NaOH + 100 cm3 H20	Temp. 50°C, czas 5 - 180 s, płukanie 5% HNO_3 i wodą lub alkoholem	Ujawnia fazy międzymetaliczne w stopach z Cu, Ni
Do cynku	Ni35Zn 5 cm3 HNO_3 (1,4) + +95 cm3 alkoholu etylowego	Temp. 20°C, płukanie alkoholem	Ujawnia granice ziaren
Do cyny i stopów	Mi38Sn 15 cm3 HNO_3 (1,4) + +95 cm3 alkoholu etylowego	Temp. 20°C, płukanie alkoholem	Ujawnia granice ziaren. Wytrawia fazy bogate w Pb
Do ołowiu i stopów	Mi42Pb 5 cm3 kwasu octowego lodowatego +95 cm3 alkoholu etylowego	Temp. 20°C, płukanie alkoholem	Ujawnia granice ziaren i struktury pierwotnej w stopach z Sb

Cross Section Polisher SM-09010

Cross Section Polisher, makes cross section perpendicullar to the specimen surface.
It is suitable for investigation of multilayer structures.

Cross Section Polisher SM-09010

Principle of operation

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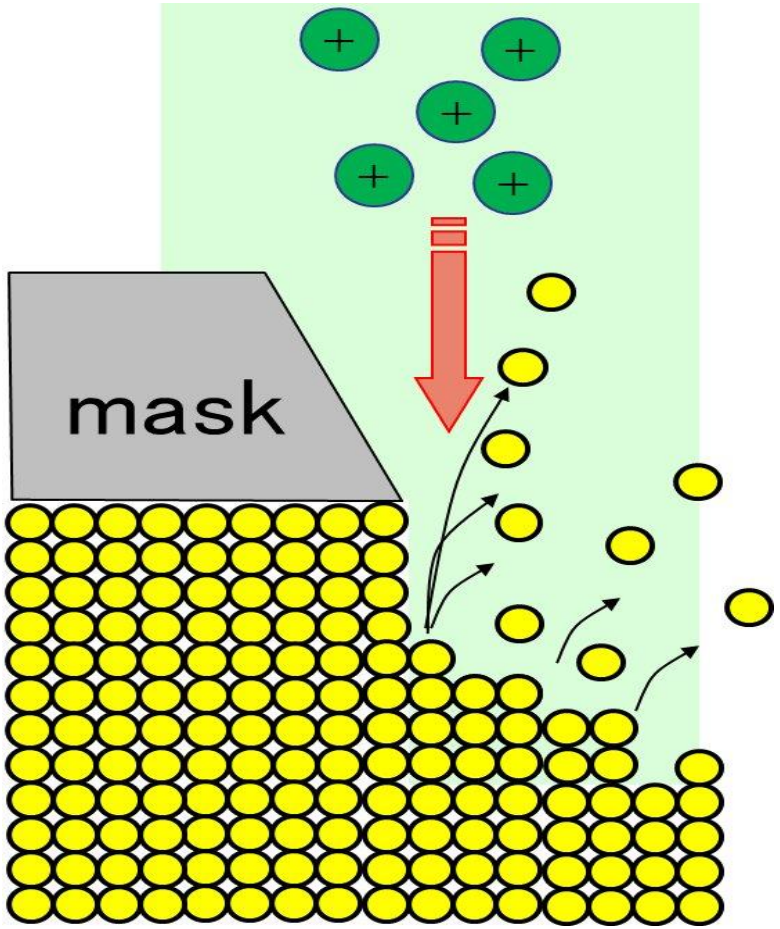
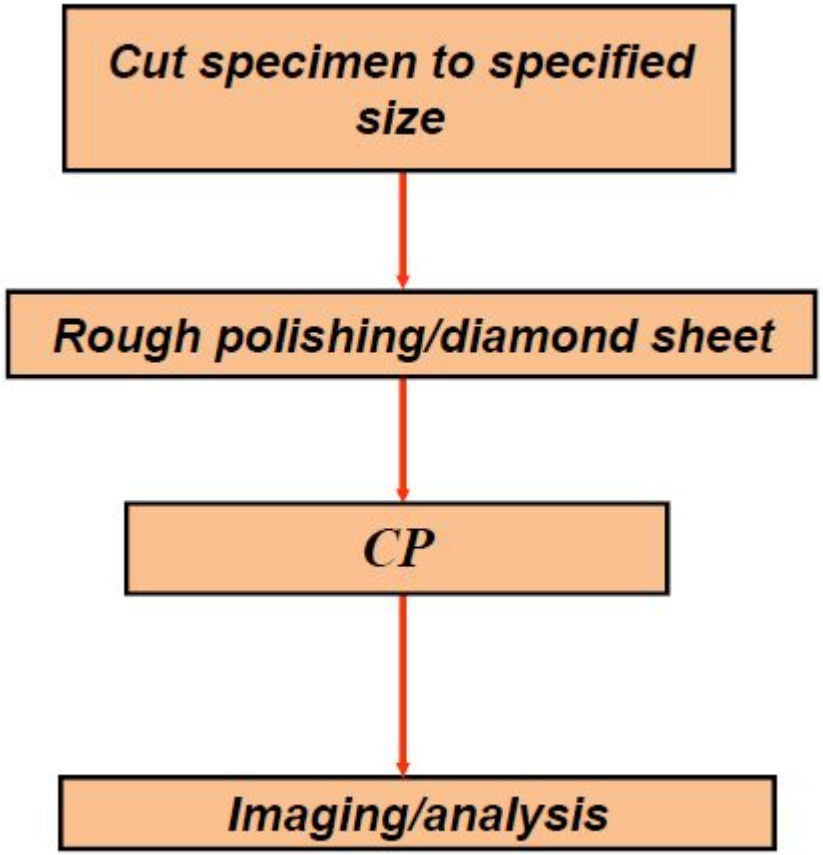
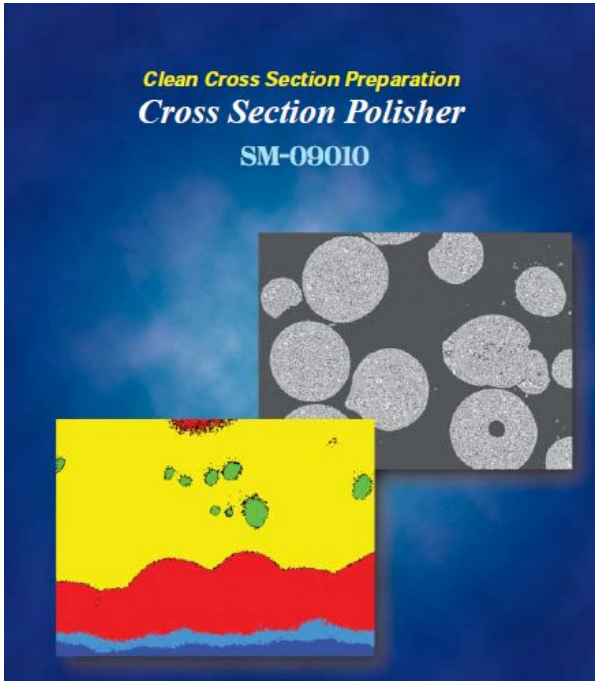


Procedura przygotowania próbki



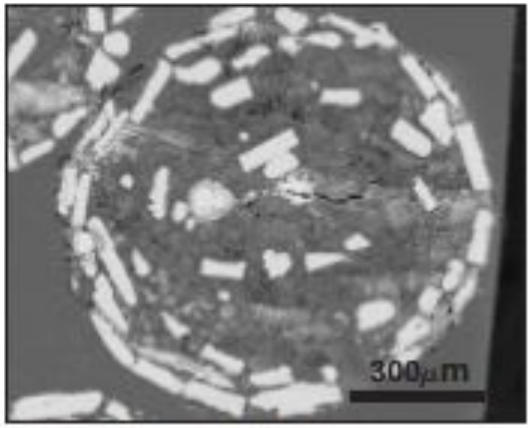


Specimens cutting

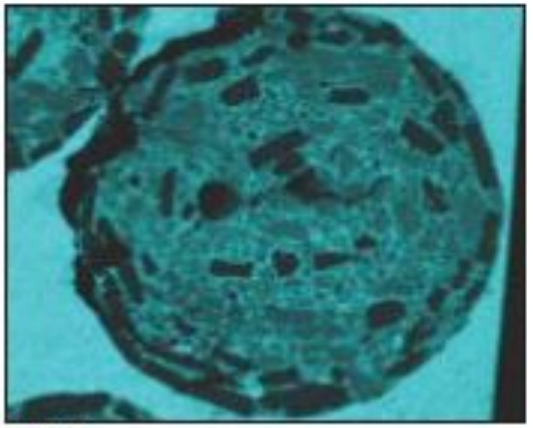
Saw equipment for sample precission cutting

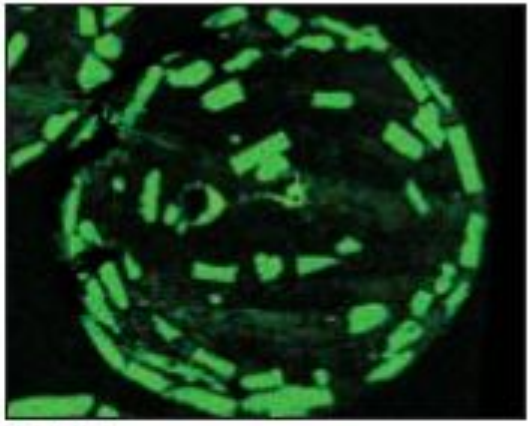
Examples of SEM application

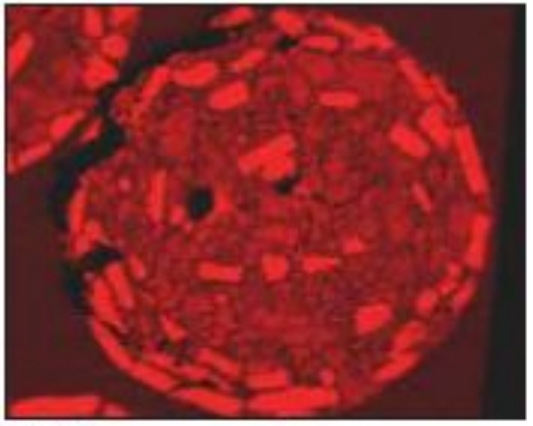
Granulated medicine



Backscattered electron image


C (EDS)

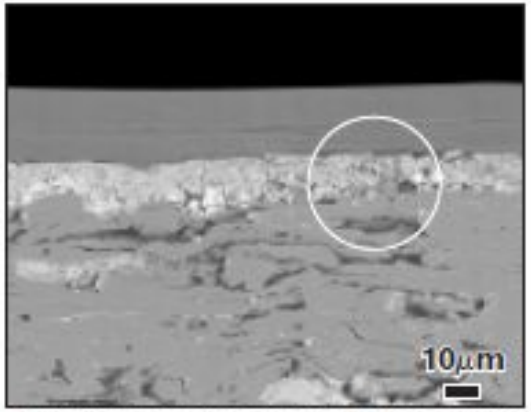

Na (EDS)


O (EDS)

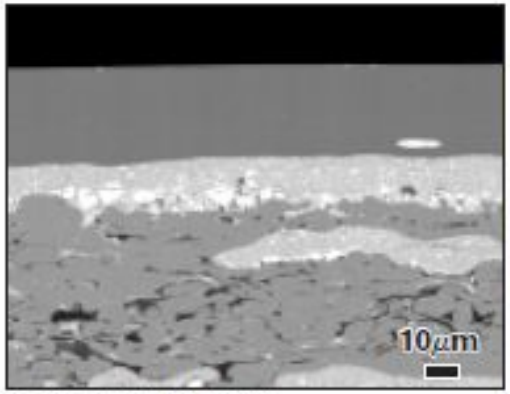
Examples of SEM application

Paper

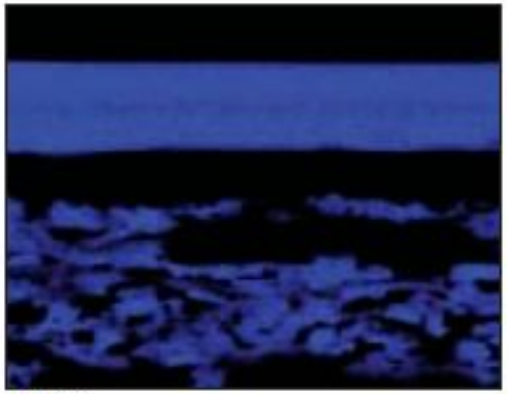
Cross-section



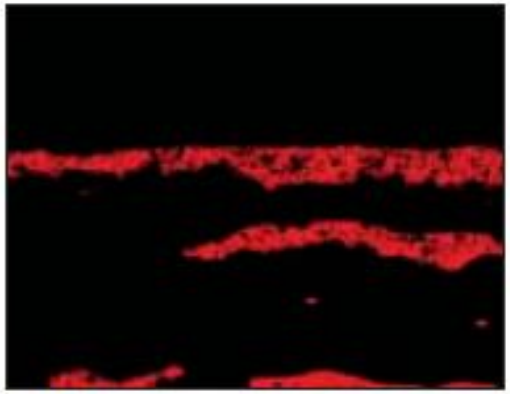
The cross section prepared with a razor edge often shows artifacts such as a loss of inorganic particles (shown in circle), or fibers being compressed.



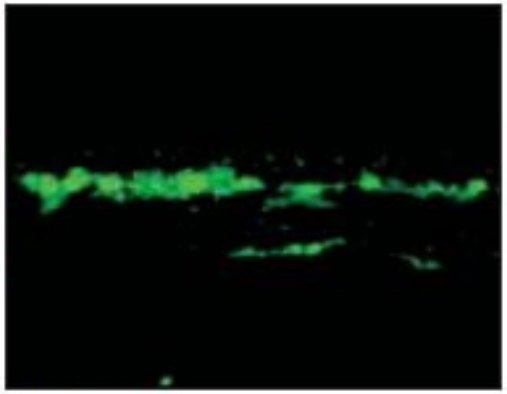
Cross section by the CP
(Backscattered electron image)



C (EDS)



Si (EDS)



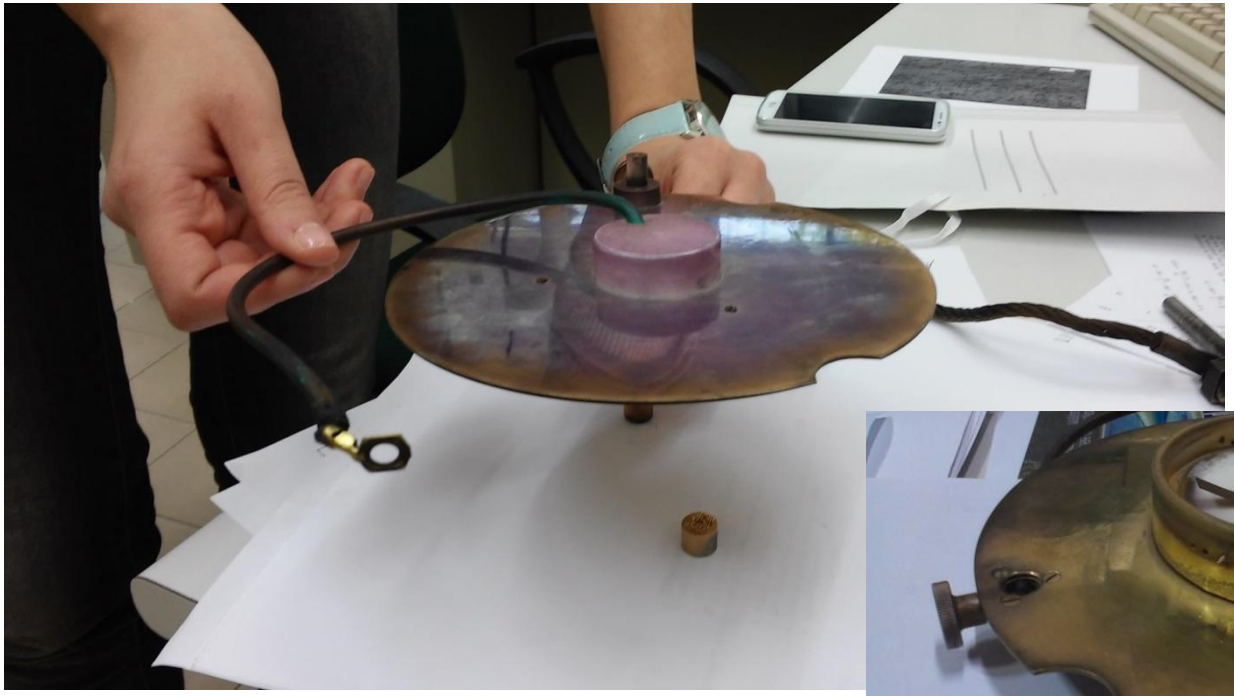
Ca (EDS)

Evaporisation / sputerring

Is realised for covering the surface specimen by C, Au, Pt or Cu at high vacuum using special equipment.

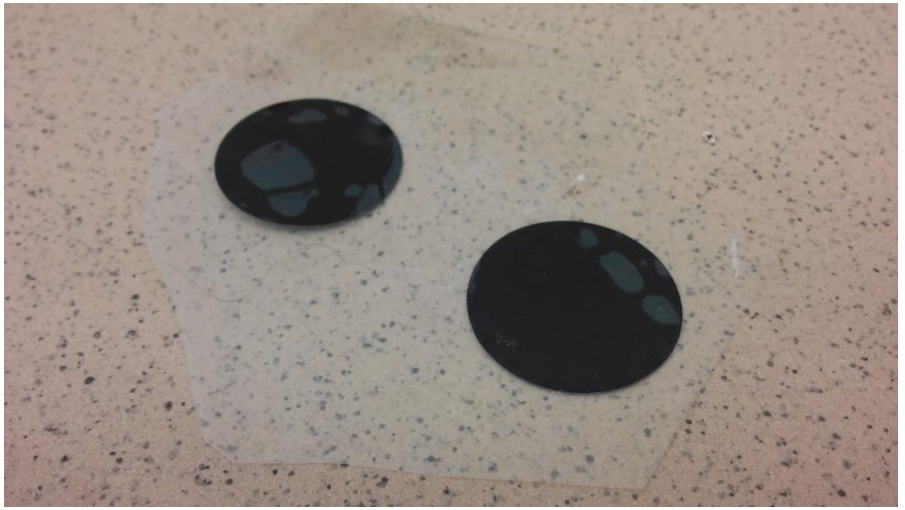
Evaporation

Cathode sputtering



Specimen fixation

Current conductive plasticine



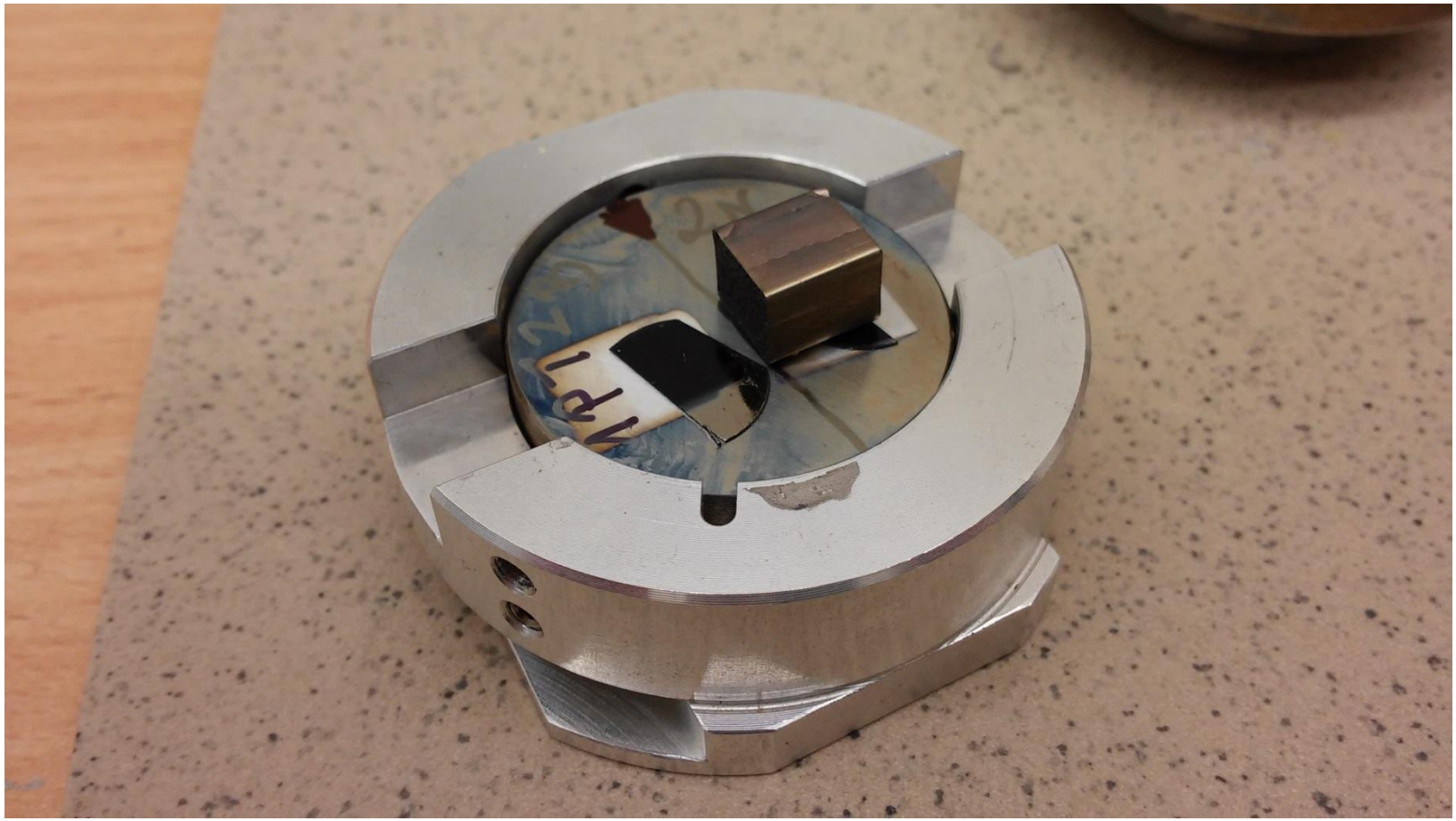
Sticky carbon discs

Specimen fixation

Silver glue

Double Scotch tape

Specimen fixed to the holder



Specimens inside the holder

Replica

The aim is to obtain direct microstructure of construction elements without their cutting or destruction.

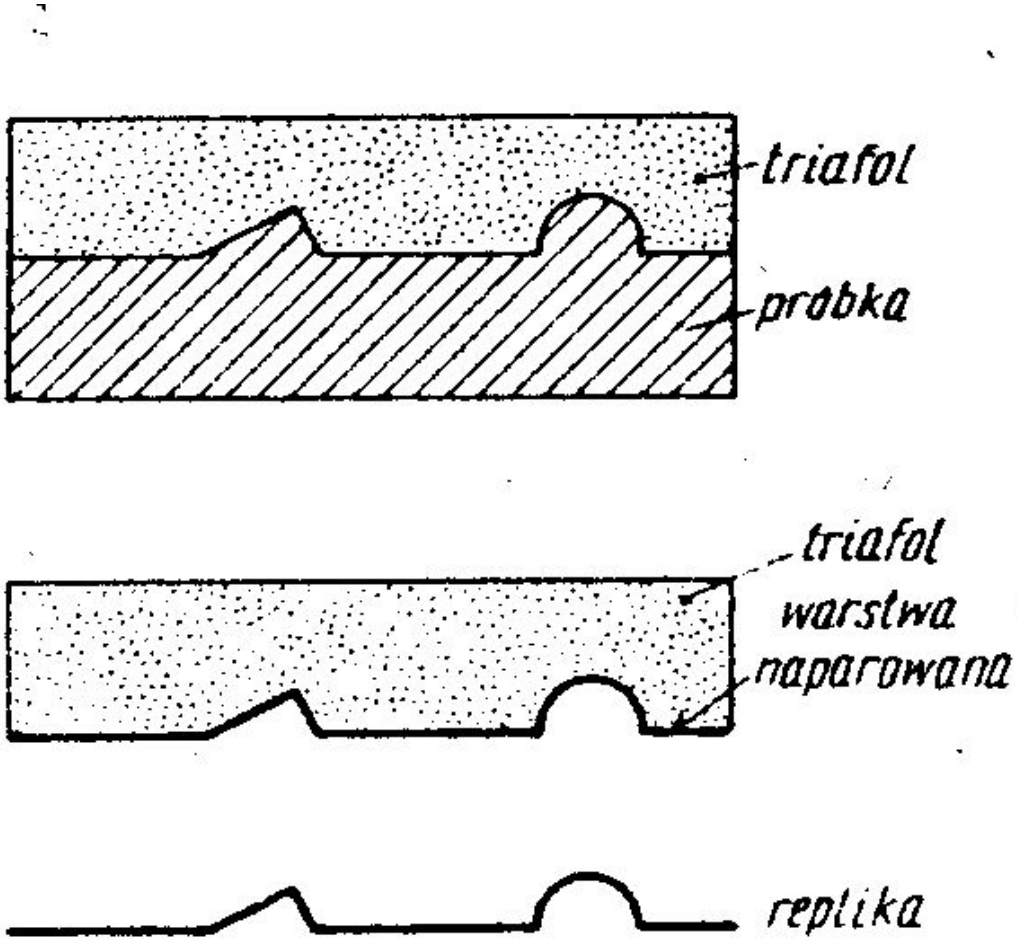
Advantages:

- Non destructive method (without decrising the strength of investigated elements).

Disadvantages:

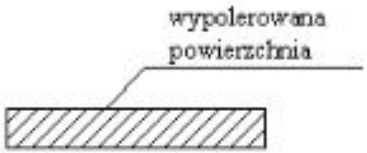
- The abbility to study only the outer surface layer (cannot be representative for whole volume / thickness of investigated material).

Replica



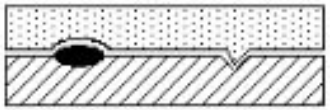
Replica

1a. Powierzchnia przygotowana do badań

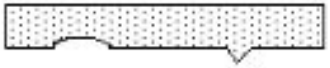


1b. Zgład trawiony

2. Replika matrycowa nałożona na zgład trawiony

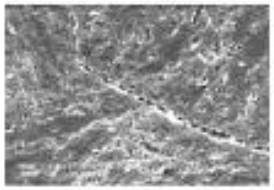


3. Replika po zdjęciu ze zgładu

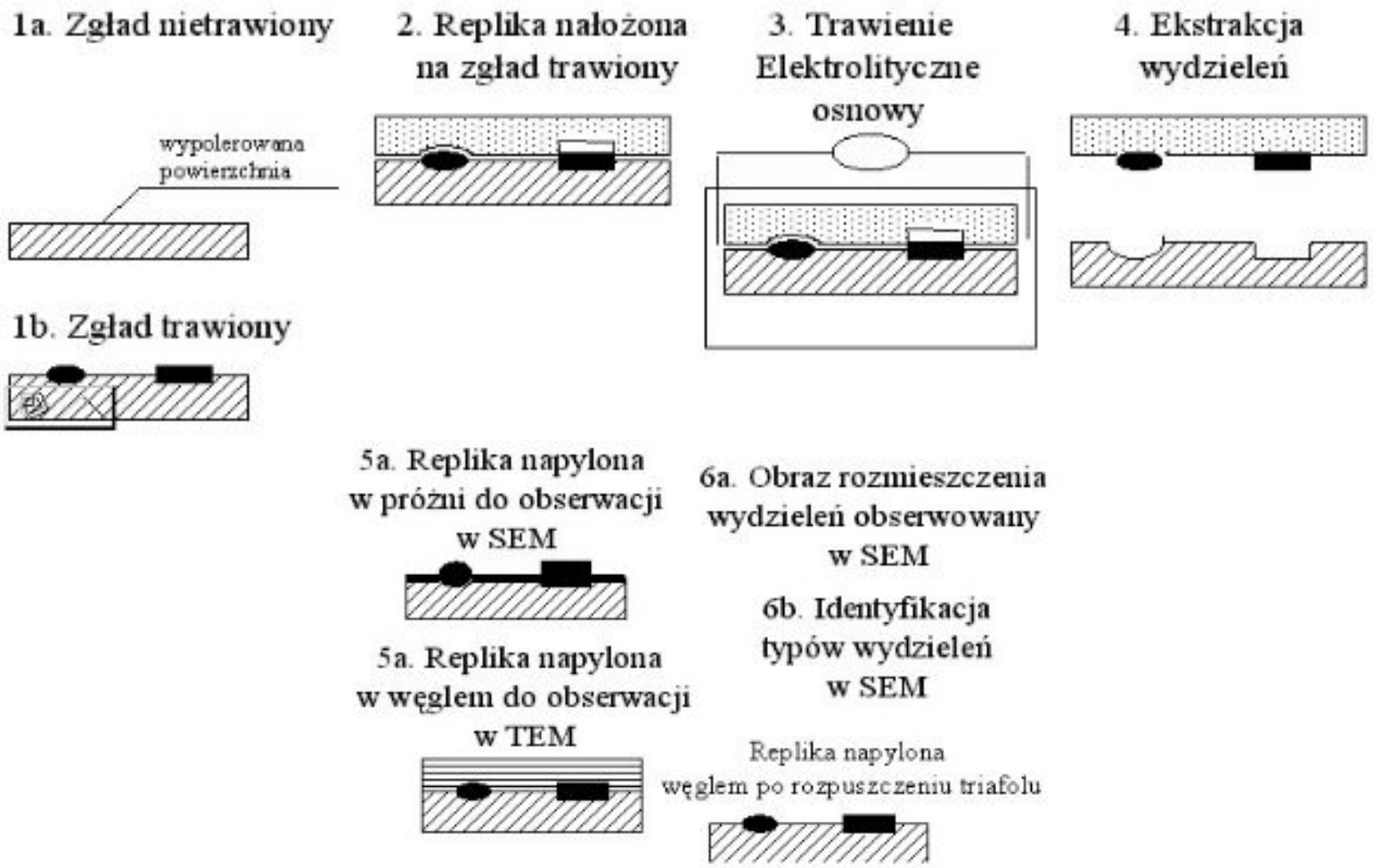


4. Replika po napylenia w próżni warstwą zwiększającą kontrast

5. Obraz struktury obserwowany w skaningowym mikroskopie elektronowym

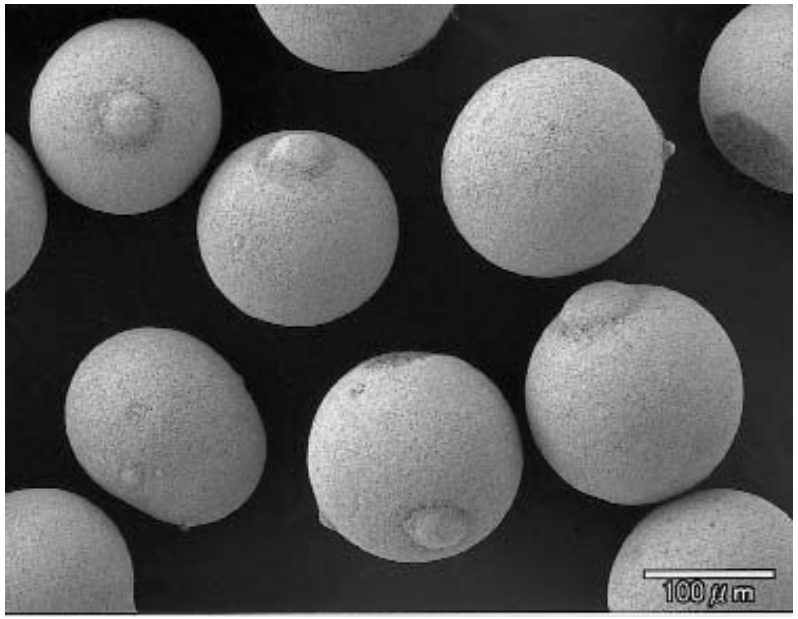


Extraction replica

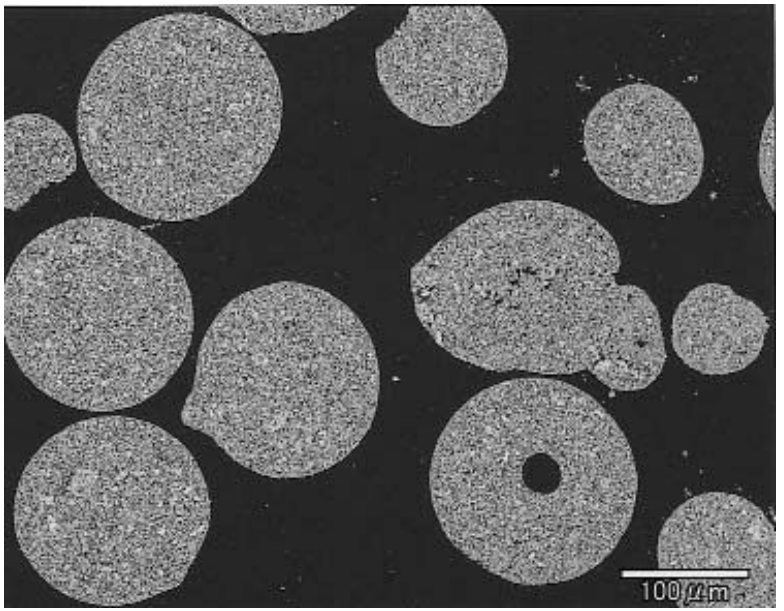


Examples

Surface of ceramic powder

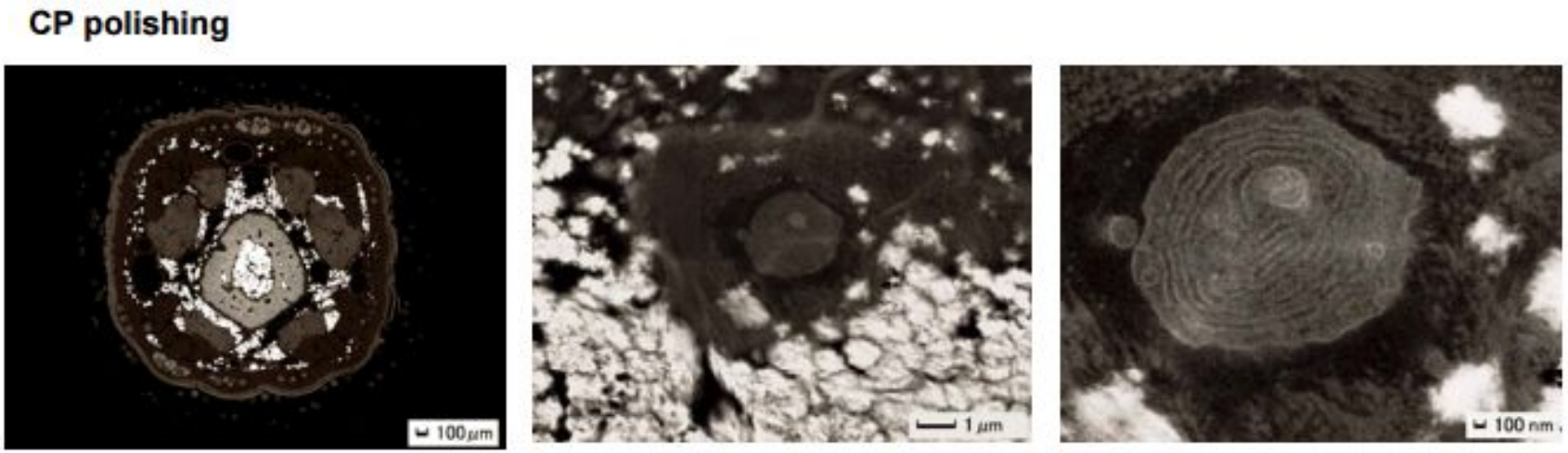


Cross section of ceramic powder

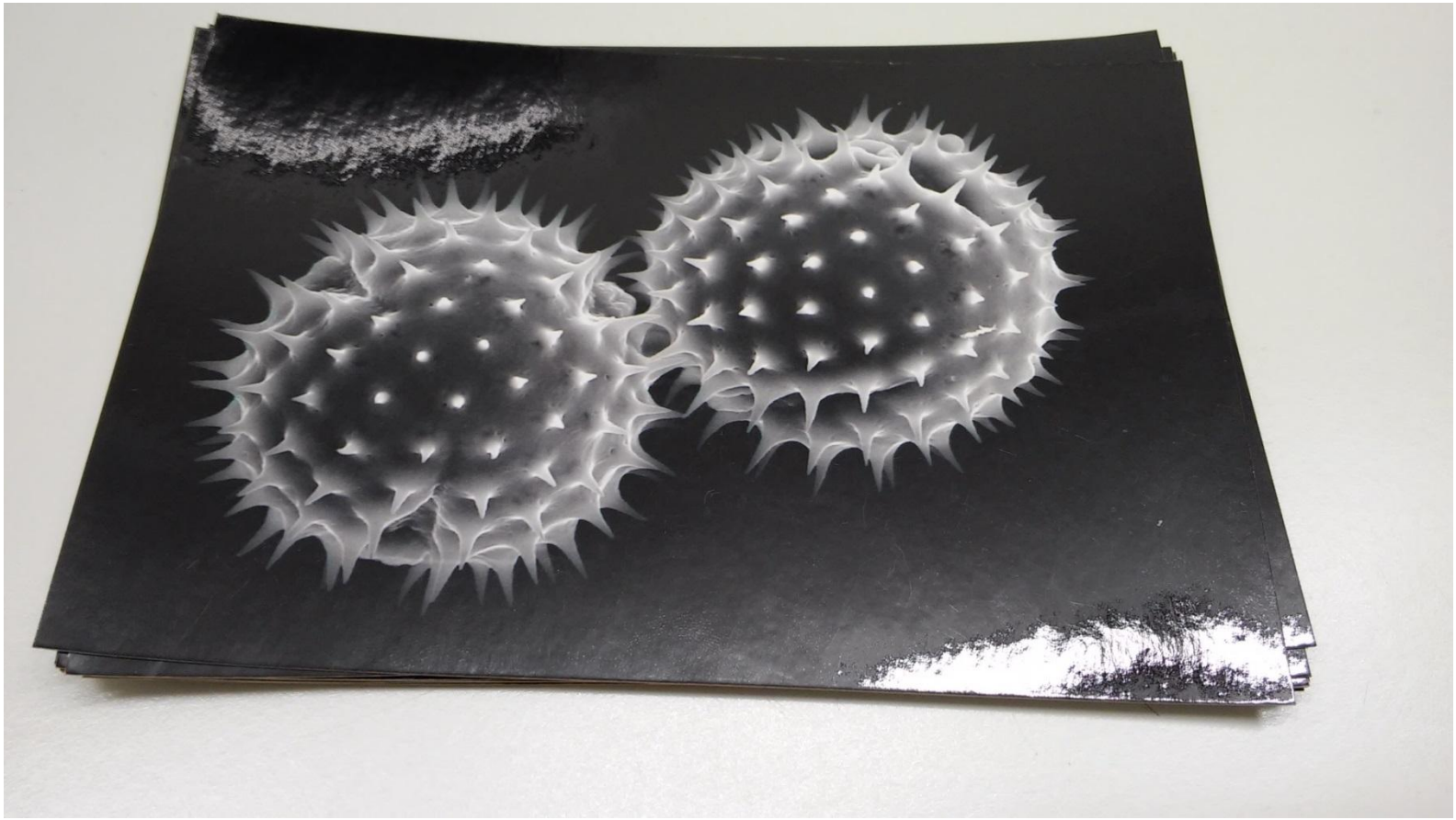


Examples

Cross section of bone



Pollen of flowers



Part of insect head

Brittle fracture

Composite