

Methods of structural analysis

Nanomaterials characterization I

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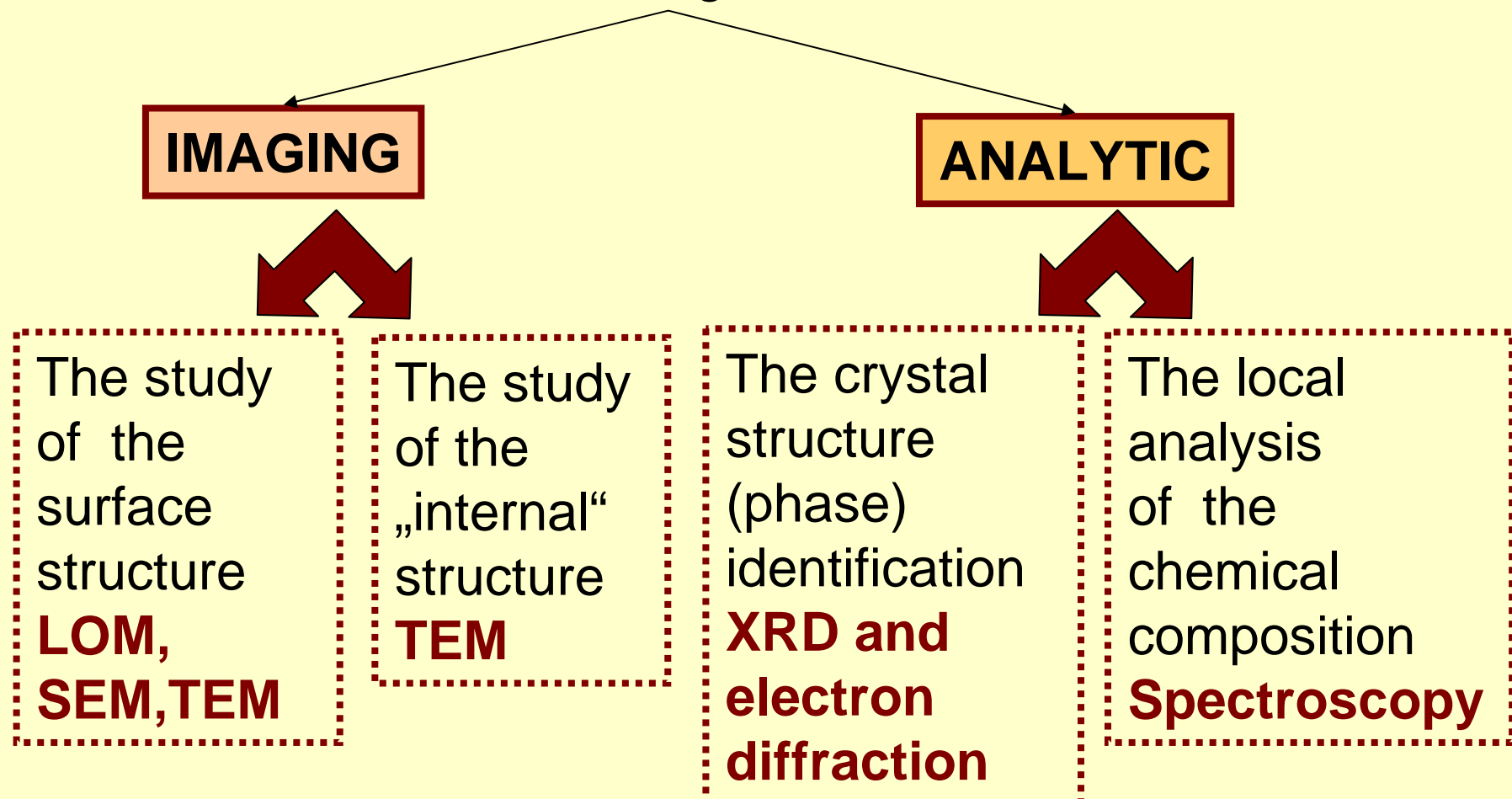
OP Vzdělávání
pro konkurenceschopnost

INVESTICE DO ROZVOJE VZDĚLÁVÁNÍ

The experimental methods used in the material engineering:

- ❖ **methods of the structure study**
- ❖ **methods of the properties evaluation**

The basic sorting of methods



Imaging methods

→The base of laboratory methods of structural analysis
The most frequently used: **microscopic methods**

➤ **light (optical) microscopy (LOM)**

The basic metallographic method (metallographic microscopy = the modification of the conventional light microscopy)

➤ **scanning electron microscopy (SEM)**

Primary probe = an electron beam; signal SE, BSE

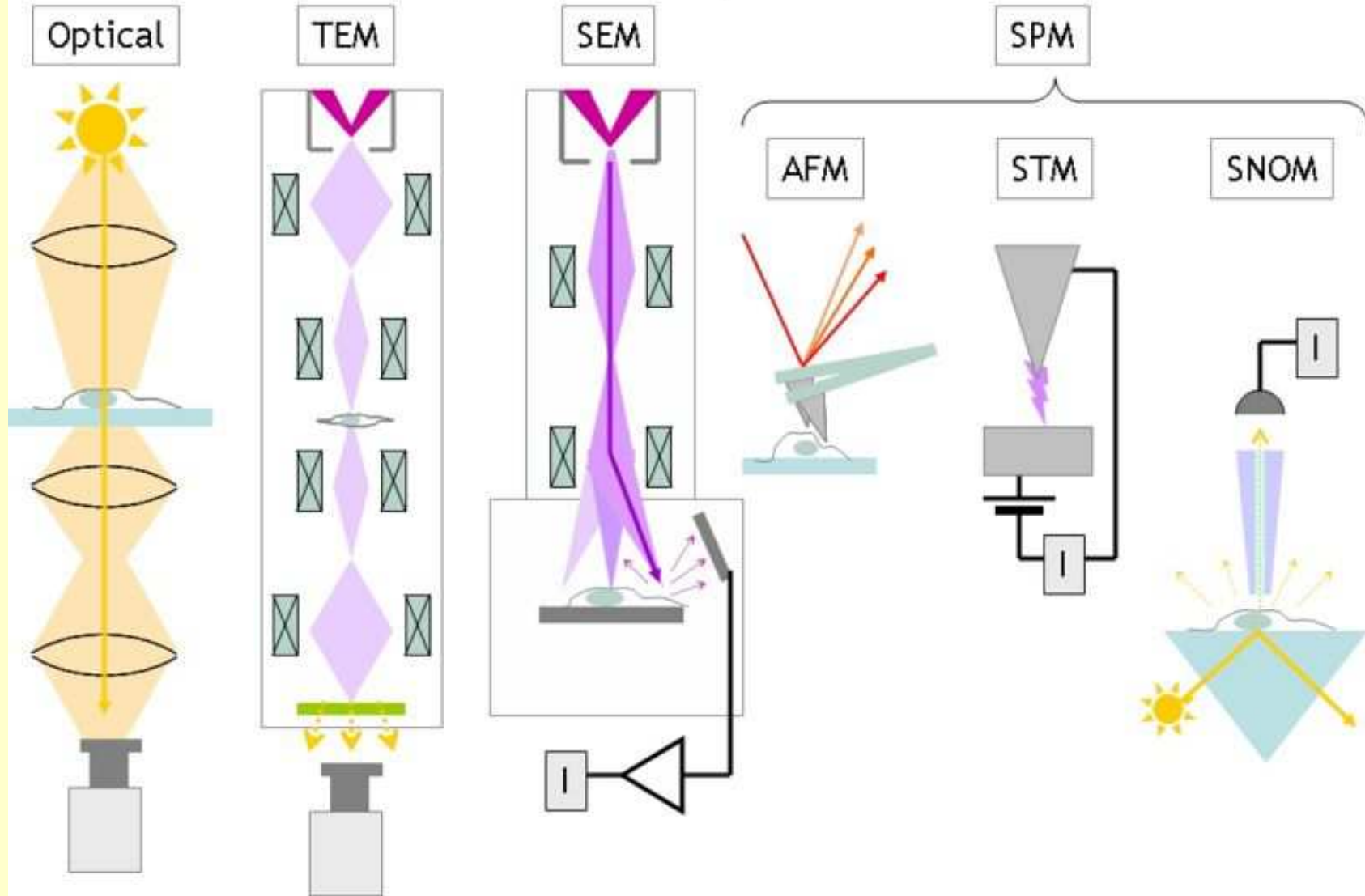
➤ **transmission electron microscopy (TEM)**

Primary probe = an electron beam; signal TE, DE

➤ **scanning probe microscopy (SPM)**

An interaction: the probe - the surface, the outstanding resolution

Microscopes



Dependence of methods option on study structure volume

Size	Typical structural characteristic	Method	
atomic $10^{-4} - 10^{-2} \mu\text{m}$	lattice defects	TEM	SPM
submicroskopik $10^{-1} - 1 \mu\text{m}$	subgrains		SEM
microscopic $1 - 10^2 \mu\text{m}$	grains	LOM	SEM
makroskopik $> 10^2 \mu\text{m}$	cracks, coarse segregates		

Another imaging methods

❑ Radiographic microscopy

- a radiation with short λ used, the magnification LOM-level
- a possibility of the internal structure observation

❑ Emission electron microscopy

- for a study of electrons emitting materials
- a specimen has the role of the cathode

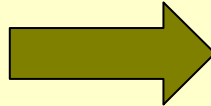
❑ Emission ion microscopy

- ions caused by a gas ionization close to the specimen surface are used for imagination
- the ionization probability \sim the ordering of atoms on the surface \Rightarrow ions created image of ordering and its defects

Analytic methods

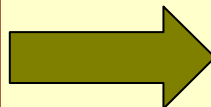
Aims of analytic methods → the determination of the chemical composition of the phase and phase identification (the determination of the crystal structure)

The type of crystal lattice and its parameters determination



X-ray diffraction methods (x-ray and electron diffraction)

The chemical microanalysis (the analysis of the chemical composition in very small volumes, the area is studied in scanning mode simultaneously)



X-ray microanalysis (exciting of characteristic X-ray using electron beam) EDA, WDA

Analytic methods comparison

Method	Analytic possibilities			
Electron diffractography	the determination of the crystal structure			
	elements	min.	size	depth of inf.
EDAX	$Z \geq 11$	10ppm	1 μm	1 μm
WDAX	$Z \geq 4$	10ppm	1 μm	1 μm
AES	$Z \geq 3$	0,10%	0,5 μm	1,5 μm