UNIVERSITY OF CHEMICAL TECHNOLOGY AND METALLURGY

FACULTY OF METALLURGY AND MATERIALS SCIENCE

COUNCIL OF MATERIALS SCIENCE

Approved by: …….……

Dean Associate Prof. Dr. I. Gruev

EDUCATIONAL PROGRAM

Subject: Microstructural Characterization of Materials
Specialization: Materials Science
Educational Degree: Master
(with teaching in English)
Qualification: Engineer

Authors:

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I. Hours, according to the educational plan

Type of occupation	Semester	Number of hours
1. Lectures	II	20

2. Practice and seminars	II	20
3. Form of control	Examination	

II. Annotation

This course is oriented to the students with interest in the field of Materials Science. The content continues the subject of the course "Structural Analysis" for students from the Bachelor degree of the specialization "Materials Science and Metallurgy". The lectures include the modern methods for microstructural characterization of bulk and thin film materials and also, the selection, application and the data interpretation of the methods.

All methods are considered, according to the character of ray-matter interaction, as follows:

- modern applications of light microscopy (LM) in the Materials Science;
- conventional and advanced electron microscopic and microprobe methods (transmission electron microscopy - TEM, scanning transmission electron microscopy - STEM, high resolution electron microscopy - HREM with TEM and SEM modifications, high voltage electron microscopy - HVEM, scanning electron microscopy - SEM, scanning tunneling microscopy - STM, atomic force microscopy - AFM and electron probe microanalysis - EPMA);
- diffraction (scattering) methods (X-ray diffraction - XRD, selected area electron diffraction - SAED, neutron diffraction - ND) and their classical and recent applications for structural analysis of crystalline and amorphous materials.

The lectures are supported by appropriate examples concerning the microstructure of selected materials. The exercises include demonstrations of microstructural analysis in leading laboratories of UCTM and some Institutes of BAS. The purpose of the seminars is to make students acquainted with the basics for interpretation of the microstructural data.

III. Lectures

1. Microstructure of materials

Microstructure - properties relationship in Materials Science. Structural levels in the materials' characterization - visual observations, as well as observations by means of LM, TEM, SEM and AFM. Scattering investigations - XRD, SAED and ND. Microstructural parameters of materials: size of structural elements (grains, microcrystals, immiscibility structures, etc.), dislocations and dislocation density, phase volume fractions in multiphase materials. ***1 hour***

2. Methods for microstructural characterization of materials.

Classification of the methods. Conventional, non-conventional and polyfunctional equipment - general principles and peculiarities. Basic parameters of the methods - precision, sensitivity, locality degree, resolution, usefull and total magnification, sharpness and focusss depth. Obtaining and of treatment of microstructural information - stages and examples. ***1 hour***

3. Light microscopy

Basis of geometrical optics. Image formation in LM - reflection and absorption of the light, bright field (BF) and dark field (DF) contrast, phase contrast. Metallographic microscope. Study of monophase and polyphase systems. Quantitative metallography. Optical anisotropy and polarized light. Interference microscopy. Examples. 4 ***hours***

4. Transmission electron microscopy (TEM).

Image formation and types of contrast in TEM analysis of crystalline and amorphous bulk and thin film materials. Coupling of TEM data at BF and DF modes with SAED results. TEM observations of dislocations. Interpretation of data from TEM analysis, artifacts. Examples. ***2 hours***

5. N on-cony entional TEM methods.

High voltage electron microscopy (HVEM). High resolution electron microscopy (HREM). Equipment peculiarities and techniques for samples preparing. Image treatment. Examples. ***1 hour***

6. Scanning electron microscopy (SEM).

Image formation and types of contrast in SEM. Equipment. Peculiarities of techniques for preparing of samples from different materials. Interpretation of data from SEM analysis for back scattered electron (BSE) and secondary electron (SE) modes. Coupling of SEM results with data from quantitative and qualitative EPMA. Examples. ***2 hours***

***7. Special probe methods* - STM and AFM.**

Physical basis of STM and AFM. Applications of methods for investigations of the atomic structure of surfaces and thin coatings for estimation of the smoothness degree, surface depositions, defects and molecular absorption. Data interpretation. Examples. ***2 hours***

***8. Electron probe microanalysis* (EPMA).**
Qualitative, semi-quantitative and quantitative EPMA with energy dispersion (EDAX) and
wave dispersion (WDAX). Equipment. Analytical electron microscopy – combination between data from EPMA and TEM, SAED and SEM analysis. Examples. ***2 hours***

***9. X-ray diffraction* (XRD).**
X-ray-matter interaction. X-ray diffractometers. Qualitative and quantitative XRD analysis of mono- and polycrystalline materials for determing the parameters of crystalline lattice and sizes of crystals. XRD analysis of amorphous materials. Radial distribution functions (RDF) of atoms and parameters of short range order (SRO) in bulk and thin film amorphous materials. Small angle X-ray scattering (SAXS) for estimation of the sizes of structural elements. Examples. ***3 hours***

10. Selected area electron diffraction (SAED) and neutron diffraction (ND) analysis.
Wave properties of electrons and neutrons. Electron and neutron scattering from crystalline and amorphous bulk materials and thin films. Kikuchi lines - interpretation. Small angle neutron scattering (SANS). Application of EXAFS for determining of the coordination number. Peculiarities and comparative analysis of the advantages and disadvantages of the scattering methods - XRD, SAED and ND. Examples. ***2 hours***

IV. Exercises

1. Light microscopy. Techniques for preparing of the samples - grinding, polishing and chemical treatment for development of the surface structure. Metallographic microscope -manipulations with eyepiece-micrometer and object-micrometer. Observation and image registration of polished samples. Qualitative and quantitative analysis in BF and DF mode of non-metal inclusions in metals and alloys. Quantitative metallography - determing of grain sizes and shape and distribution of secondary phase. ***6 hours***

2. TEM of crystalline and amorphous bulk and thin film materials. Techniques of samples' preparing for direct and indirect TEM observation. BF and DF modes in TEM. TEM analysis of dislocation structure of materials. SAED of crystalline and amorphous materials. Registration and interpretation of TEM images. Software treatment of TEM data for determining of size distribution of particles and structural elements. Examples. **4** ***hours***

***3. Scanning electron microscopy (SEM) and electron probe microanalysis* (EPMA).**
Techniques for preparing of the samples, observation, image registering and data interpretation from SEM and EPMA of metals, alloys, semiconductors, ceramics, polymeres and composites. Examples. ***6 hours***

***4. X-ray diffraction* (XRD) analysis.**

Obtaining and interpretation of XRD data from crystalline materials. X-ray scattering from amorphous materials - interpretation of RDF of atoms for obtaining the parameters of the SRO. Examples - inorganic, organic and metal materials. ***4 hours***

V. References

1. Количественный электронно-зондовой микроанализ, под ред. В. Скот, Г.Лов, Москва, Мир, 1986 /превод от английски/.
2. Дифракционные и микроскопические методи в материаловедении, под ред. С. Амелинкс, Р. Геверс, Дж.Ван Ланде, Москва, Металургия, 1984 /превод от английски/.
3. Analyse structurale et chimique des materiaux, Jean-Pierre Eberhart, Dunod, Paris, 1997.
4. Физические методы исследования в неорганической химий, И. М. Жарскии, Г. И. Новиков, Москва, Высшая школа, 1988.
5. Science of Materials, T.J. Lewis et al., Gerge Harrap & Co, LTD, 1966./
6. Microstructural Characterization of Materials, D. Brandon and W.Kaplan, John Wiley & Son, LTD, 1999.
7. Основи сканирующей и зондовой микроскопии, В. Миронов, Техносфера, Москва, 2004.
8.Рентгенография металлов, Я. С. Уманский, Москва, Металургия,1967.
9. Структура металлов, Ч.С. Баррет, Т.Б. Массальский, Металлургия, Москва, 1984 г.
10. The structure of non-crystalline materials - Liquids and Amorphous Solids, Y. Waseda, Ed. McGraw-Hill Inc., USA, 1980.