

Európska únia Európsky sociálny fond





Applicant:	Slovak University of Technology in Bratislava
Project:	Knowledge-based Faculty for Economic Practice
ITMS code of project:	26110230113
Activity:	3.2
Responsible for activity:	PhDr. Kvetoslava Rešetová, PhD.

# THE INSTITUTE OF MATERIALS – PROFILE PRESENTATION

Name of activity	Activity 3.2 Building tools for knowledge transfer into training
Name of specific aim	3. Building tools for knowledge transfer into training
Aim of activity	The activity of building the tools for knowledge transfer into education concludes the information flow attained from the analysis of environment impact, and sets up specific tools for the knowledge transfer into education. It is aimed at building a set of tools for transferring the knowledge collected in the previous activities into education at the Faculty, thus enhancing the knowledge base of the target group, while focusing on the functionality of the knowledge transfer to the target group.
Date of activity implementation	10/2013 - 09/2015

## Project part: activity 3.2 : 1. Tool for knowledge transfer – profile research presentations

The Faculty research is oriented particularly on the following fields:

- research in materials with focus on the research, development and technological processing of the main types of engineering materials,
- research and development of new technologies in industrial production oriented mainly on technological processing of modern technical materials and environment-friendly production,
- research in identification, automation and control of processes as well as information security of the technology, production and organizational systems,
- research and verification of principles of managerial control and its organizational structures,
- · research in quality and certification of processes and products,
- research in safety and reliability of technological devices and systems with emphasis on the methods of systems analysis and synthesis.

The defined research characteristics reflect the Faculty research fields, and are subject to the Faculty evaluation processes. The current profile presentations comprise the research profile, its identification and recording in a new way. The Faculty experts in the research fields make the audience familiar with the research characteristics, research and development orientation, so that to provide a comprehensive research profile of the Faculty institutes (there are six institutes at the Faculty) in both Slovak and English languages. The elaboration of text in an adequate number of quires, text translation and copyright rules – all this is subject to the method of the profile presentation implementation. The elaborated profile presentations might be an important material source for:

- training at the Faculty in its key subjects
- domestic and international presentation of the Institute
- enhancement of the advertising space for promotion purposes of the project
- innovative elaboration of the Faculty research contents.

# Defined project outcomes:

The project outcomes will be determined by successful implementation of the project activities, particularly activity 1.1 - stakeholding, activity 2.1 - portal of companies, activity 3.1 -implementation from acquired e-. sources. a realizacie aktivity 3.1 - z získaných e ... Such interaction along with the information flow can influence the success of knowledge transfer into education. The outcomes of previous activities will be utilized in this final activity which should provide space particularly for knowledge transfer and improvement of knowledge base, and simultaneously provide a space for meeting the main project aim. Specific outcomes of the activity will be as follows:

- six profile presentations mapping the research character of six Faculty institutes, applicable in training and with strong potential for the Faculty promotion
- production of minimum 30 virtual records of technological procedures outsourced from economic practice and applicable in education, i.e. enhancement of information on applicability for the Faculty doctoral students
- production of minimum 30 virtual records of the Faculty technological procedures and processes, for application in the Faculty education, and for the purposes of comparison of the technological processes and theoretical knowledge acquired in the Faculty training to the knowledge acquired in practice

 four expert lectures for doctoral students (and also for interested Faculty researchers), forming the knowledge base of the target group in four principle science fields.

# Implementation of activity:

- 1. In compliance with the project aims, the activity was introduced to the Heads of the STU MTF Institutes: **Appendix 1**: Information for institutes of 12 Dec 2013 , **Appendix 2** : Letter to the Heads of the STU MTF Institutes of 21 Jan 2014
- Heads of the STU MTF Institutes delegated in writing an Institute representative who will be in charge of the profile presentation elaboration – letters to Heads of the Institutes are in the project archive of the principle investigator
- 3. Individual meetings of the principle investigator with related employees with focus on structure and contents of profile presentations
- 4. Collection of data, text modifications, graphical design of presentations
- 5. Text translation
- 6. Final arrangement of presentations into e-proceedings of scientific papers
- 7. Publicizing the profile presentations

# Guarantors of profile presentations:

Institute of Materials - Mgr. Marián Palcut, PhD.

Institute of Production Technologies - Assoc. Prof. Ing. Erika Hodúlová, PhD.

Institute of Industrial Engineering and Management – Assoc. Prof. Ing. Helena Makýšová, PhD.

Institute of Safety, Environment and Quality - Prof. Ing. Maroš Soldán, PhD.

Institute of Applied Informatics, Automation and Mechatronics – Prof. Ing. Pavol Tanuška, PhD.

Advanced Technologies Research Institute – Assoc. Prof. Ing. Maximilián Strémy, PhD.

The orientation of STU MTF research activities comes out from the Faculty profile in pedagogy and is in compliance with the long-term development of the Slovak University of Technology in Bratislava and covers the all spectrum of the education at STU MTF. The activities of STU MTF researchers are implemented as follows:

- projects of base research supported by VEGA grant agency,
- projects of applied research supported by KEGA grant agency,
- projects investigated within international programmes,
- projects of international scientific and technical cooperation,
- projects of base and applied research supported by APVV grant agency,

contractual research and development (business contracts).

## 1. Science and Research in STU MTF

#### Vision of STU MTF

The STU Faculty of Materials Science and Technology in Trnava, in compliance with the STU vision, intends to be a research oriented and internationally renowned faculty within the similar faculties framework, i.e. the faculties developing modern trends in research and industrial production with focus on progressive materials, sophisticated production technologies and industrial management, automation and IT implementation of production and technological processes such as quality, safety, as well as environmental and managerial aspects of industrial production.

## Mission of STU MTF

In compliance with the defined mission of the Slovak University of Technology, the STU Faculty of Materials Science and Technology intends to actively contribute to meeting the requirements of the mission – with the priority laid on materials science and production technologies – in accredited fields of education, research and development within the stipulated competences:

- provide the university system of education in all stages in accredited study programmes
- disseminate, improve and develop knowledge by the research and development tools,
- ensure transfer of research results into educational process,
- ensure transfer of research results into entrepreneurial practice,
- protect its research results,
- integrate into the system of university life -long learning,
- participate in sustainable development of society with all its activities, mainly by the development of the student personality in the context of humanism and democracy ideals.

## General and strategic goals of research

- Publish the research and creativity results internationally, particularly in the renowned international scientific journals.
- 2. Increase the STU MTF status in the projects of international cooperation.
- 3. Build the research infrastructure (equipment) including the qualified service.
- 4. Intensify the cooperation with practice, ranging from private industrial companies to public institutions and authorities.
- 5. Focus the research results and free investigation also on the outcomes, e.g. patents.
- 6. Improve the orientation on other than grant sources from the state budget, particularly on the sources from abroad, project grant agencies and entrepreneurial activity.

## The scientific and research activity of STU MTF is carried out in the forms of:

- projects of the base and applied research and development,
- projects solved within the international programmes,
- projects of the international scientific collaboration,

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• projects of contractual research.

The research content is focused on the following areas:

- materials research with a focus on the research, development and technological processing of the basic and advanced types of technical materials,
- research, development and optimisation of the new technologies of industrial production, oriented
  particularly on the technological processing of advanced technical materials and ecologically clean
  processes and products, and the numerical simulation of technological processes,
- process identification, automation and control, as well as information support for technological, production and organisation systems,
- research and verification of the managerial control principles and their organisation structures,
- quality control and certification of processes and products,

safety and reliability of technological equipment and systems, while emphasising the methods of system analysis and synthesis.

#### 2.1 Institute characteristics

The STU MTF Institute of Materials was founded on 1 January 2007 by merging three original departments and thereby building qualitatively better opportunities for research activities and international cooperation.

The Institute provides the training of materials and physics oriented subjects at STU MTF. On the Bachelor degree study it guarantees Materials Engineering study programme, on Master and Doctoral study degrees the Institute guarantees the following study programmes – Materials Engineering, Processing and Application of Non-metals as well as Surface Engineering of Advanced Materials. The Institute prepares the experts in materials engineering with bachelor, master or doctoral degrees required in production companies and research institutes.

In the pedagogical, research and expertise activities the Institute focuses on complex materials analysis of metals, alloys, plastics, glass and ceramics. The research and expertise activities are oriented on crystallization of metals and alloys, tool materials and nickel-based alloys, powder metallurgy, bio-compatible materials, stainless steels, steels for power industry, welding properties of steels, magnetic materials, heat processing and surface finishing of materials, complex metal alloys, grain boundaries engineering as well as ceramic and plastic materials.

Currently, the Institute has seven laboratories at its disposal. In the laboratories there are several modern experimental devices available (e.g. Philips CM300 – high resolution transmission electron microscope, Philips PW 1710 - X-ray diffractometer, Perkin Elmer – differential scanning calorimeter). In research as well as in its pedagogical activities the Institute closely cooperates with national and international institutions and is the important component of the wide network of international academic as well as commercial workplaces thus providing possibilities for students and teachers exchange. The Institute successfully cooperates with internationally renowned production companies such as Böhler-Edelstahl, Bekaert, etc., universities (POSTECH, TU Vienna, Austria, Ljubljana University, Slovenia, etc.) and research institutes (IFW Dresden, FZD Rosendorf, Germany, IJS Ljubljana, Slovenia, etc.). The most significant are as follows: Leibniz Institute for Solid State and Materials Research Dresden (Germany), Institute Jožef Stefan, Ljubljana (Slovenia), Vienna University of Technology (Austria), Research Centre Dresden-Rossendorf (Germany), Institute of Materials Physics of Czech Academy of Sciences, Brno (Czech Republic), Faculty of Mechanical Engineering, University of Ljubljana (Slovenia), as well as some Slovak universities and several Slovak Academy of Sciences Institutes. The most important foreign manufactures are: Bekaert SA, Böhter - Edelstahl and Branson div. Emerson.

The Institute of Materials has long term contacts with regional enterprises including for example, INA SKALICA, spol. s r.o. Skalica; VUJE, a.s. Jaslovské Bohunice; ZF Sachs Slovakia, a.s., Trnava; Zlievareň (Foundry), a.s., Trnava; HKS Forge s.r.o Trnava; MANZ, a.s. Nové Mesto nad Váhom; SONY Slovakia, Nitra; Samsung Electronics Slovakia, Galanta, Voderady; Faurecia Trnava; PSA Peugeot Citroen, Trnava; Noble International, spol. s r.o. Senica; TRW Steering System Slovakia spol. s r.o., Nové Mesto nad Váhom; Hella Lighting Slovakia, Kočovce; Kinex-KLF, a.s., Kysucké Nové Mesto; PSL, a.s. Považská Bystrica; EMO, a.s. Mochovce; Johns Manville, a.s. Trnava; Sauer Danfoss, a.s., Považská Bystrica; ŽOS a.s., Trnava; PFS, a.s., Brezová pod Bradlom; Kompozitum Topoľčany; Fremach, Trnava; Slovalco, a.s. Žiar nad Hronom; IMS Kupa, a.s. Nováky.

#### 2.2 Institute of Materials - Centre of Excellence

The Institute of Materials is the investigator of significant international and national projects. An extraordinary significance of the Institute's research is represented by

# Centre for Development and Application of Advanced Diagnostic Methods for Processing Metal and Nonmetallic materials

The main aim was to build the centre of Excellence with focus on the development and application of diagnostic methods for processing the metal and non-metallic materials within the 2.1 measure of the Operational Programme Research and Development *"Quality Improvement of Research Workplaces and Support of Excellent Research with Emphasis on the Fields with Strategic Importance for Further Economy and Society Development"*. In these terms the main aim was to build the research infrastructure in compliance with the 2nd Generation Innovation policy, i.e. on regional level and in compliance with No. 1 Priority of the Innovation Policy Platform of the Slovak Republic: *"High Quality Infrastructure and Effective System for Innovations Development"*.

The project was aimed at building the modern dynamic Centre of excellent analytical methods utilizing current latest knowledge in the interaction of electron and laser volumes with the mass, advanced detection high sensitive systems, modern mechanical procedures and monitoring of electrical and non-electrical quantities focused on the evaluation of specific properties mainly progressive metal and non-metallic materials prepared by latest technological procedures. The project was oriented on the support of concentrating the best (excellent) Faculty experts into a one centre based on the application of the latest experimental procedures characterizing the specific materials properties regarding the Materials study programme and scientific branch Physical Metallurgy. The activities were focused on attracting the secondary school students for the study of technology and material oriented study (at present there is a regional project guaranteed by STU MTF and financed by Trnava city which supports this kind of activities), on making the modern tool technology of the centre available to the experts interested, on organizing seminars and summer schools as well as promoting the material research and its successful representatives in media.

The Centre is equipped by modern technology:

- Scanning high resolution microscope with thermal FEG cathode complemented by the file of cooperating detection EDS, WDS a EBSD systems with related devices for sample preparation via ion milling.
- Laser confocal microscope with two independent laser volumes for wave lengths of 400 and 600 nm.
- Universal testing machine for mechanical properties evaluation of metal and non-metallic materials
- Testing device for monitoring of the failure in process dynamics
- Apparatus for measurement of alternative conductivity of non-metallic materials at higher temperatures
- Spectral analyser allowing the measurement of impedance and modular spectrums of non-metallic materials and composites
- Rotation viscometer
- Vulcanograph

#### Annotation of Centre of Excellence Project

1	
Názov projektu	Centrum pre vývoj a aplikáciu progresívnych
	diagnostických metód v procesoch spracovania
	kovových a nekovových materiálov
Name of project	Centre of development and application of
	progressive diagnostic methods for processing metal
	and non-metallic materials
TMS projektu / ITMS of project	26220120014
Doba riešenia / Duration of project	05/2009-04/2011
Pracovisko riešenia / Workplace	Ústav materiálov
	Institute of Materials
Operačný program / Operational programme	OPVaV - 2008/2.1/01-SORO

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Anotácia

2

Projekt je zameraný na vybudovanie moderného dynamického centra excelentných analytických metód, ktoré využívajú súčasné najmodernejšie poznatky interakcie elektrónového a laserového zväzku s hmotou, špičkové detekčné systémy s vysokou citlivosťou, moderné mechanické postupy a sledovania elektrických a neelektrických veličín. Zameriava sa na hodnotenie špecifických vlastností progresívnych kovových a nekovových materiálov, ktoré sa pripravujú najmodernejšími technologickými postupmi. Ciele projektu sledujú vybudovanie moderného diagnostického centra hodnotenia vlastností kovových a nekovových materiálov, vypracovanie nových postupov metodiky zameriavajúce sa na využitie moderných analytických prístrojov, na zisťovanie štruktúrnych, mechanických a elektrických vlastností materiálov v procese ich výroby. Annotation

The project is aimed at building a modern dynamic Centre of excellent analytical methods utilising the current advanced knowledge on the interaction of electron and laser beam with matter, top detection systems of high sensitivity, modern mechanical procedures and monitoring the electric and non-electric quantities. It is focused on evaluating the specific properties of progressive metallic and non-metallic materials prepared by the advanced technological procedures. The project goals comprise the building of a modern diagnostic centre for evaluating/assessing the properties of metallic and non-metallic materials, elaborating new procedures and methods for utilising advanced analytical devices, and identifying structural, mechanical and electric properties of materials in the manufacturing process.

Názov projektu	Centrum excelentnosti pre vývoj a aplikáciu
	diagnostických metód pri spracovaní kovových a nekovových materiálov - APRODIMET
Name of project	Centre for Development and Application of
	Advanced Diagnostic Methods in Processing of
	Metallic and Non-metallic Materials
ITMS projektu / ITMS of project	26220120048
Doba riešenia / Duration of project	01/2010-12/2011
Pracovisko riešenia / Workplace	Ústav materiálov
	Institute of Materials
Operačný program / Operational programme	OPVaV - 2009/2.1/02-SORO
Anotácia	

Hlavné zameranie projektu sleduje rozšírenie prístrojového vybavenia moderného dynamického centra excelentných a analytických metód využívajúcich súčasné najmodernejšie poznatky z interakcie rtg. žiarenia

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s hmotou, moderných postupov merania a hodnotenia mechanických, termofyzikálnych a koróznych vlastností progresívnych kovových a nekovových materiálov a špičkových termodynamických detekčných systémov s vysokou citlivosťou, moderných postupov spracovania povrchových vrstiev a hodnotenia efektu interakcie rôznych fyzikálno-chemických účinkov na ich životnosť a exploatačné vlastnosti. Realizáciou projektu sa vytvorilo päť ucelených laboratórnych celkov: Laboratórium tepelných tokov, Laboratórium termofyzikálnych meraní, Laboratórium koróznych skúšok, Laboratórium štruktúrnych analýz a Laboratórium povlakovania a tepelného spracovania.

## Annotation

Project is focused on enhancing the equipment of a modern dynamic centre of excellent and analytical methods. It will utilise the latest knowledge of X-ray interaction with materials, along with advanced procedures of measuring and assessing mechanical, thermo-physical and corrosive properties of progressive metal and non-metallic materials, top thermodynamic detection systems of extra sensitivity and advanced procedures of processing the surface layers. The centre will provide the assessment of various physical-chemical effects on the lifetime and exploitation properties of metal and non-metallic materials. The project implementation gave rise to five complex laboratory units: Laboratory of heat flows, Laboratory of thermo-physical measurements, Laboratory of corrosion tests, Laboratory of structural analyses and Laboratory of coating and heat treatment.

# 2.3 Institute of Materials – Research fields

#### 2.3.1 Corrosion (by Mgr. Marián Palcut, PhD.)

The term "corrosion" describes the various electrochemical phenomena occurring on the materials surface and driven by external conditions such as increased temperature, atmospheric pressure, various chemical agents in the solution, etc. These processes can lead to a gradual material mass loss and subsequently induce its degradation.

In the STU MTF Institute the corrosion of materials is investigated. At present, the Institute coordinates VEGA Project focused on the study of zinc, aluminium and tin alloys corrosion. The studied materials can be applied as light construction materials for automotive and aviation industry, for steel coatings or lead free solders for microelectronics.

Common practice shows that individual metals are subject to corrosion to different degrees. This is because of different electrical potentials of these metals after immersion in a solution. Therefore, from the practical point of view, the metals are arranged into a reactivity series according to the values of their standard electrode potentials. The metals in the upper parts of the series have positive potential values. These metals are called noble metals as they are rather corrosion resistant. They are as follows: silver, gold or platinum. On the other hand, the metals on lower levels of the series are less noble as they are more corrosion susceptive. They include e.g. zinc-, aluminium- or tin-based materials.

If two different metals are connected and immersed in an aqueous solution, one can learn that an electric current runs between them. The voltage between two connected metal materials is established as a result of different electrical potentials of individual metals. The connection of two metals immersed in the solution is called a galvanic couple. The principle of the galvanic couple is used in the study of corrosion. In the corrosion study a galvanic couple of the studied material and a reference electrode is built. Under given experimental conditions the reference electrode has a precisely known electric potential. In the study of corrosion the tested material is being polarized. During polarization the electric potential of the measured metal is varied and the

resulting electric current is monitored in the circuit. For the electric potential regulation and measurement of the current running through the electric circuit the potentiostatic device is utilized.

The result of the study of electrochemical corrosion of metals is represented by polarization curves. The curves show whether the given metal corrodes regularly, irregularly or whether it is able to resist the corrosion under specific conditions.

Despite the fact that some metals, e. g, aluminium or magnesium are not noble materials, they can resist corrosion under certain conditions due to their passivation ability. In passivation a thin layer of oxides, hydroxides or other corrosion products is built on the metal surface and they protect the metal from further oxidation. On the polarization curve the property is indicated by a sudden current decrease.

Galvanic couples can be built also within one material if this material comprises structural heterogeneities or various phases. For instance the corrosion of Al-Co-based alloys is interesting. These alloys comprise several phases with different nobility. When studying the corrosion of these alloys one can learn that that the phases with low cobalt concentration corrode preferentially in sodium chloride water solution. The corrosion activity of the phases might be influenced by their chemical composition. Cobalt is more noble metal than aluminium as it has a higher electrode potential. The phases rich in cobalt are therefore more corrosion resistant. The co-existence of phases with various chemical compositions supports the origin of galvanic micro-couples which in many cases are the driving force of corrosion.

The study of materials corrosion is very important particularly from the practical point of view and helps increase the materials lifetime. The study of corrosion combines the knowledge in several natural sciences, mainly physics, chemistry and materials engineering. The Institute places particular emphasis on the combination of experimenting and modelling. The necessary tool infrastructure for the STU MTF Institute of Materials has been acquired via European structural funds within the Centre for Development and Application of Advanced Diagnostic Methods in Processing of Metallic and Non-metallic Materials. The Institute has also ThermoCalc SW operating on Calphad principle at its disposal as well as many databases allowing predicting the phase composition of investigated alloys. The mutual synergy of experiment and thermodynamic modelling of alloys allows a development of existing databases and improves materials property predictions. The combination of experiment and modelling is beneficial as it makes this workplace comparable to similar research institutions abroad.

#### 2.3.2 Thermal analysis (by Ing. Marián Drienovský, PhD.)

Netzsch STA 409CD Simultaneous Thermal Analyser is determined for simultaneous analysis of thermal effects and mass changes in a material during the set temperature regime with the possibility of analysing the released gas products. Used methods are indicated as differential thermic analysis – DTA, differential scanning calorimetry – DSC. Thermogravimetry – TG, derivation thermogravimetry – DTG, and the analysis of evolved gas via mass spectrometry – EGA-MS. The measurements can be carried out in the air atmosphere or in inert gas, e.g. Ar, He.

The device is capable to record the phase changes in a material such as melting temperature, temperature of lattice formation, or temperature of magnetic transfer (Curie temperature). At the same time, the sample mass can be observed as function of temperature (e.g. for oxidation, absorption, reduction, decomposition, sublimation or evaporation of the sample). In addition, via the mass spectrometer it can analyse the chemical

composition of released gas molecules from the sample, or analyse the products of its decomposition and hence identify the material.

Technical data of simultaneous analyser NETZSCH STA 409CD: Temperature range of measurements: from room temperature to 1,600 °C Speed of heating or cooling: 0.1 to 30 °C/min Atmosphere: oxidation (synthetic air 80/20), or inert (argon, helium, nitrogen) Gas flow: from 0 up to 150 ml/min Mass of sample: max. 15 g Temperature difference DTA and DSC: 0.1°C Sensitivity of scales by thermogravimetry: 2 µg Range of mass spectrometer measurement: 1 to 512 atomic mass units Mass spectrometer sensitivity: 1 ppm

STA applications in practice and material research are mainly as follows:

- measurement of thermal capacity of materials
- determination of temperatures and enthalpies of phase changes
- analysis of kinetics of phase changes
- determination of magnetic transfers/transits
- observation of processes related to oxidation, reduction, absorption, adsorption, sublimation and evaporation
- analysis of thermal stability and material decomposition
- identification of decomposition products via mass spectrometer
- determination of materials purity

#### **Figures description**

#### Fig. 1

The first figure shows DSC record of the prepared sample. The sample was placed in a device in which it was gradually heated from the room temperature up to 450°C (red curve). Then there was cooling again to the room temperature (blue curve). Unless the sample does not change its state in the course of heating or cooling, the device records a straight line. As soon as the sample starts to melt during the heating (or solidifies during the cooling), the device records a peak, or a set of peaks representing the melting or solidification process of the material. This way it is possible to state the melting temperature of the tested alloy with accuracy. As seen in the record this specific alloy of tin, copper, indium and silver does not melt at a constant temperature but in the temperature interval from 211.7°C up to 336°C. The device can also measure the power necessary for melting the sample. In this case it is 74 Joules for each gram of the alloy. As seen in the record, solidification of the alloy does not run at the same temperatures as its melting. This effect is called sub-cooling of the melt and can be from a few degrees up to several tens of degrees at high speed cooling.

## Fig. 2

The figure shows a combined DTA + TG record of a small sample of K390 tool steel. In this case the sample was heated from the room temperature up to 1100°C. I tis important to know that the sample was subject to magnetic field created by permanent magnets during the whole measurement time. The red curve illustrates the change of properties as well as the change of sample structure at the temperatures given. The first peak at 784°C is connected to a small change of mass at the same temperature – green curve. This peak represents the loss of magnetic properties in the material, from this temperature the sample stops to be attracted by magnetic field. The temperature of this effect is called Curie temperature and in general it occurs in all iron-based alloys. The other peak is at 832.7°C represents the change in the structure of the crystal lattice of the steel given. This temperature should be known especially in heat processing of steels.

#### Fig. 3

The last figure shows the simultaneous DTA-TG-MS record of Ch12MF4 powder steel. This powder sample was heated in the protective atmosphere from the room temperature up to 1400°C. The black curve is DTA record, green curve represents the mass of the sample during the analysis and the red curve is the flow of particles released from the sample into the mass spectrometer. The mass spectrometer allows identifying of specific gas molecules, or specific particles sublimed or evaporated from the sample (blue peaks). Indicated temperatures of 674.2°C and 859.9°C on DTA curve are phase changes of the sample in its solid state. At the temperature of 1227.4°C the material begins to melt. As seen from the figures, during the heating nitrogen and carbon oxide release from the powder surface. It is due to the fact that the powder was prepared by spraying the melt in nitrogen atmosphere. After the powder melts carbon oxide starts to evaporate intensively and similarly the heavier elements such as vanadium, chromium or iron. Carbon oxide probably occurs in the steel during the reaction of carbon with residual oxygen in protective atmosphere.

## 2.3.3 Scanning electron microscopy (by Ing. Marcela Pekarčíková, PhD.)

Nowadays, the scanning electron microscope represents an inevitable part of material analysis. In contrast to microscopes utilizing visible light for microstructure observation, in electron microscopes the highly accelerated electrons are used. This will ensure substantially better resolution ability of the scanning electron microscope which is from 2,000 to 1,000,000 times better that the one of a human eye. The Institute's laboratory of structural analyses possesses JEOL JSM7600F – a high resolution scanning microscope with the resolution of several nanometres at the accelerating voltage of 15kV. The electron beam directed and focused by the set of electromagnetic lens in vacuum scans the sample surface similarly as the beam in TV screens did. By the fall of the primary electron beam onto the surface of the tested material it comes to mutual interaction where the primary electrons can strike off, therefore, we call them backscattered electrons, or new secondary electrons can release from the sample during the interaction. Via four detectors the secondary and backscattered electrons are collected, converted into electric impulses and processed by computer into 3D representation of the sample surface. The scanning electron microscope is equipped by spectrometers for energy- and wave-dispersive X-ray analyses and diffraction of backscattered electrons via which it is possible to obtain information on chemical composition and crystal orientation of individual grans in the sample.

The main advantage of the microscope is that it requires no demanding sample preparation and it can be also used for observation of non-conductive or special materials, e.g. ceramic superconductors based on metal oxides of rare earths, barium and copper which are superconductive, i.e. without electric resistance at extremely low temperatures, however, at the room temperature they behave as insulants.

Within the VGA project the STU MTF Institute of Materials also deals with (RE)BCO superconductors which are ones of the most frequently used materials with superconductivity at quite high temperatures, e. g by minus 180°C. The research focused on detail structure analysis of only 1.5  $\mu$ m thin superconducting layers deposited on a metal substrate allows understanding the relation between the conditions of layer preparation and its resulting properties. During the deposition of (RE)BCO layer on a metal tape as well as in further processing various structural inhomogeneities can occur in the superconducting layers. These inhomogeneities have a significantly negative influence on electromagnetic properties of superconductors because its strong required grain orientation – so called biaxial texture – is disrupted. The obtained information about damaged structure of (RE)BCO layers can be utilized for decreasing the error rate in the production process of superconductor layers. The knowledge is also necessary for the design of superconductor devices as they can significantly influence their operational characteristics.

## 2.3.4 Transmission electron microscopy (TEM) (by Assoc. Prof. Ing. Mária Dománková, PhD.)

TEM is often characterized as a typical invention of 20th century. Its construction was not based on one genius idea, it was constructed due to a series of inventions of many scientists as well as due to the technological progress:

- in 1897 it was the electron discovery described by J.J. Thompson,
- in 1925 Luis de Broglie described that electron is not only a fast moving particle but electromagnetic oscillation as well,
- in 1927 this is confirmed by Davisson and Germer and Thompson and Reid by the electron diffraction which proved the wavelike behaviour of electrons,
- in 1926 H. Busch published important contributions on electron deviation due to the magnetic field of a coil and light via glass lens,
- specific concept of the possibility to construct the transmission electron microscope originated at the University of technology in Berlin – Knolle and Rusko.

The first TEM was constructed in 1932. At present, TEM belongs to significant experimental methods for detail study of technical materials. This experimental device has several advantages thus making it an irreplaceable part of experimental methods in new technical materials development.

The value of the resolution ability which is from  $10^{-10}$  m, and the high range of magnification up to 1 500 000x, allows studying the materials on their atomic level. Another big plus is represented by the possible application of electron diffraction for identification of the phases in the analysed materials.

The disadvantage of this experimental method is complicated preparation of the samples. They have to be very thin. Their width should not exceed 200 nm. In general, there are two basic groups of samples used in:

 <u>replicas</u> – they are thin amorphic layers (most frequently of carbon) perfectly copying the prepared metallographic surface. Their width is from 5 to 50 nm. The replicas enhance the field of classical metallography into higher magnifications and better resolutions. They can provide us with the information on precipitations of secondary phases, grain morphology, etc.

2. <u>thin foils</u> – represent real thinned technical material to the width of less than 200 nm. Since it is a real thinned material, all structural attributes comprised in the sample can be observed (e. g. precipitates, dislocations, vacancies, stacking faults, monitoring of orientation relations, etc.). Obviously, the observed objects are limited by the resolution ability of the microscope.

In our Institute, there are two TEMs: analytical TEM JEOL 200 CX with accelerating voltage of 200 000 V with the resolution around 10 nm, and high resolution TEM Philips 300 CM with accelerating voltage of 300 000 V and with resolution around 10<sup>-10</sup> m. Both microscopes are utilized for research-related experiments investigated at the STU MTF such as:

- 1. substructure study of duplex steels welded joints,
- 2. study of oxide dispersion strengthened ferritic steels,
- 3. study of secondary phases precipitation in various alloys (e.g. high strength steels, duplex stainless steels, austenitic stainless steels, titanium alloys, nickel super alloys, etc.)

## 2.3.5 Mechanical tests (by .....)

Technical materials are usually used for manufacturing the machines, devices, tools, equipment and constructions. The property of the material is defined as its behaviour in certain conditions. In mechanical tests we monitor how the given material behaves due to the external mechanical load.

Static tensile test is the basic mechanical test, currently being the most widely spread method for evaluating and comparing the mechanical properties of construction materials. The tensile testing machine is called a shredder. The tensile testing principle is in the mechanical tensile loading of the bar being tested. The tensile force slowly and gently grows until the tested bar is torn. During the test the machine records the prolongation in dependence on the tensile load. The recorded work diagram can provide four basic mechanical properties: tensile strength yield, shear yield, ductability and contraction. In addition, software calculates the voltage in real time as a force acting on the tested bar cross- section. Thus, we can obtain the diagram of deformation dependence.

By bending the solid it comes to the combination of tension and compression stress. By the static bending test we monitor the bending of the tested solid in dependence on the loading force. The test is primarily determined for brittle materials such as alloys, ceramics or wood. We look for maximum force by which the tested solid is damaged. The result values depend not only on material's properties but also on the way of tested solid placement. In standing position the solid can resist higher loading force.

To evaluate the materials against the brittle fracture the impact-resistance test is used. It is carried out on the device called Charpy hammer. It is a dynamical test where in on the shuttle hammer we can investigate the power necessary for breaking the dented testing bar. The bend size dependence on the force is recorded from the tension meter located on the hammer's top. The record indicates the way of the rupture spread and is mainly utilized for various materials comparison.

The work necessary to break the testing bar is tested also at very low temperatures. Via the approximation of measured data we can draw Vidal curve of transition temperature. Inflexion point of Vidal curve indicates the transmission temperature. It is the temperature by which the ductile fracture rapidly changes into a brittle one. By low temperatures the material tends to be easily fractured.

To complement the values of mechanical properties the hardness tests are used. The hardness can be defined as material resistance against the strange solid penetrates into the tested material surface. In hardness test we force in the intender into the material surface by a defined force. The harder the material, the less the intender penetrates the tested material surface. The hardness is then measured by the indentation size. The harder the material, the smaller indentation appears. It is the non-destructive method by which the material is not impaired. The new components can be tested this way as well.

Hardening is the process of heat processing aimed at material hardness improvement. The process begins by the material heating and its stay at the austenitization temperature. Subsequently, the material is taken out of the furnace and is cooled quickly in immersing into oil. Thus, the inhomogeneous martensitic structure can be obtained which means material's hardness improvement. Immediately after hardening the process of tempering should follow, this means heating onto a lower temperature in order to remove the post-hardening tension.