40 LET ELEKTRONSKE MIKROANALIZE

40 YEARS OF ELECTRON PROBE MIKROANALISIS

Po letu 1965, ko se je v Sloveniji začel hitrejši razvoj jeklarske tehnologije, je začela hitreje naraščati potreba po večjem poznanju procesov in reakcij, od katerih sta bili odvisni kakovost in cena metalurških proizvodov. Pokazala se je potreba po napravi, ki bi omogočala dovolj natančno kemijsko analizo mikrometrskih sestavin mikrostrukture različnih zlitin, npr. nekovinskih vključkov v jeklih in segregacij, njihove odvisnosti od temperature nastanka in od temperaturnega režima procesa pretvorbe litih v tržne proizvode. Raziskovalno okolje na tedanjem Metalurškem inštitutu (MI) in v delu industrije je bilo nekoliko seznanjeno z možnostmi nove naprave, saj so bili leta 1958 na mednarodni konferenci v Portorožu, ki so jo priredili MI, Max Planck Institut für Eisenforschung iz Düsseldorfa, Nemčija, in Institut de Recherches de la Siderurgie iz mesta St. Germanin-en-Laye, prvič zunaj Francije predstavljeni rezultati analize segregacij, ki so bile izvršene na taki napravi. Naprava na tem inštitutu je bila tudi uporabljena pri raziskovanju, ki je bilo potrebno za pripravo doktorata in magisterija dveh raziskovalcev MI. To je bila zadostna podlaga za odločitev Odbora za raziskave pri tedanjem SOZD Slovenske železarne, da se za MI nabavi elektronski mikroanalizator. K projektu je bilo pritegnjenih še nekaj inštitutov, nekaj sredstev pa je prispeval tudi tedanji Kidričev sklad, in v začetku leta 1969 je na MI začela delo prva elektronska mikrosonda na Balkanu in med prvimi v srednji Evropi.

Interes za različne analize je hitro rastel med raziskovalnimi inštitucijami in industrijskimi podjetji iz cele Jugoslavije, in v prvih petih letih dela je bilo vsako leto za analize vzorcev, ki niso bili iz MI, porabljenih povprečno 1200 ur. Analize so bile izvršene za metalurška podjetja s področij jekla, sive litine, zlitin aluminija, bakra in dragih kovin, zlitin za elektroniko in zobno protetiko, različnih stekel in keramičnih materialov, pa tudi za raziskovalce, ki so delovali na področjih geologije, kemije in fizike trdnega stanja. Intenziven razvoj metodologije je omogočil, da se je dosegla analitska ločljivost, ki je bila večja od tiste, ki jo je zagotavljal dobavitelj naprave, npr. 0,001 % S v trdni raztopini v feritu, dovolj natančna analiza vsebnosti natrija v steklu, čeprav se je zaradi segrevanja pod udarom curka elektronov koncentracija tega elementa v točki analize hitro zmanjševala in točna analiza materialov, ki niso prenesli udara majhnega curka elektronov.

Prej kot leto po začetku dela naprave so bila v znanstveni in strokovni periodiki tiskana prva dela s podlago na rezultatih elektronske mikroanalize, kako leto kasneje pa tiskana tudi v periodiki v drugih državah.

After 1965, in Slovenia the development of steel technology became faster and a better knowledge of the processes and reactions that affected the quality of metallurgical products became more urgent. There was a need for a device that would make possible an accurate chemical analysis of micrometer-size details of the microstructure of different alloys, for example, non-metallic inclusions in steels, segregations in steels, aluminium and copper alloys, the investigations of their behaviour during the processing of cast products to final products, the understanding of the effects of composition, temperature and time on the microstructure and properties and a sufficient knowledge of the basic problems of materials science and technology. The research and development employees in the Metallurgical Institute (MI) and in part of industry were given some information about the possibilities of a new developed device, as in 1958 in Portorož an international conference was organised by the institutes MI, Max Planck Institut für Eisenforschung (MPIE), Düsseldorf, Germany and Institut de Recherches de la Siderurgie (IRSID), St. Germanin-en Laye, France and the results of the analyses of the segregations in steel were presented for the first time outside of France. The device in IRSID was also used in the investigations carried out for a doctoral and a master thesis of two MI scientists. These were solid reasons for the decision of the research board of the company SOZD Slovenian Ironsworks to install at the MI an electron probe microanalyser. The project was joined by some institutes, and financial contributions were also obtained from the public Kidrič Foundation and in the beginning of 1969 the first electron-probe microanalyser in the Balkans and one of the first in Central Europe started to work at the MI.

The interest for analyses increased rapidly in research institutions and industrial companies from the whole of Yugoslavia. In the first five years of analytical work, on average 1200 hours were used for analyses outside the mother institute by different research institutions and industrial companies involved in research or producing steels, grey irons, copper and aluminium alloys, precious alloys, alloys for electronics and dental prosthetics, different glasses and ceramic materials and for scientists active in the topics of geology, solid-state chemistry and physics and dental prosthetics. The rapid development of analytical methodology allowed us to achieve a very high chemical sensitivity (0.001 % S in solution in ferrite), an accurate analysis of sodium in some glasses in spite of the local decrease of content caused by the electron analytical beam impacting on the examined point and the proper analysis of materials with a low stability under the impact of the electron beam. Already after the first year of use, the first articles with the results of the electron-probe analyses were published in national scientific and professional journals and some years later in foreign journals, also.

Brez pretiravanja lahko trdimo, da je ta analiza omogočila tudi pospešen razvoj proizvodne tehnologije jekla, bakrovih in aluminijevih zlitin, rast kakovosti proizvodov teh industrij in razvoj novih proizvodov; pripeljala je do izvirnih dosežkov raziskovalnih in tehnoloških spoznanj, npr.: kinetika homogenizacije segregacij v jeklih, v bakrovih in aluminijevih zlitinah, natančna sestava nekovinskih vključkov v jeklih, kvazibinarni ravnotežni sistemi, narava in vzroki poškodb jeklenih cevi v termocentralah, nov mineral, izvirna spoznanja o bolezenskih poškodbah zob in druga.

Utemeljen je sklep, da je začetek uporabe elektronske mikrosonde pospešil tehnološki razvoj na področju metalurške industrije, pozitivno vplival tudi na keramične tehnologije in omogočil nova znanstvena spoznanja, ki so raziskovalce iz Slovenije uveljavila tudi v mednarodnem prostoru. Način nabave in dela elektronske mikrosonde bi bil lahko vzor, kako je mogoče novo raziskovalno napravo nabaviti, razviti metodologijo dela in jo izkoristiti v majhnem okolju, da imata od nje korist raziskovalna in industrijske sfera.

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It can be concluded, without any overstatement, than electron-probe microanalysis helped the rapid development of the industrial technology of steel, copper and aluminium alloys, the growth of the quality of products from these metallic materials and also allowed us to obtain original findings of international scientific and technological importance, for example, the kinetics of the homogenisation of segregations in steels and copper and aluminium alloys, the accurate composition of non-metallic inclusions in steels, the diffusion rate in the Ni-W binary system, the quasi-binary equilibrium systems of iron with VC, TiC and NbC carbides, the nature and the cause of failures of high-pressure steel tubing in thermal power works, the discovery of a new mineral, original findings on teeth lesions, etc.

The conclusion that the results of the electron-probe microanalyser in the Metallurgical Institute after the year 1969 had a beneficial effect in Slovenia on the development of the processes of metallic materials technology, production, hot working and heat treatment, an increase of the products' homogeneity and quality, also had a positive effect on the development of ceramics and enabled us to achieve original scientific findings that were important for different materials and their industrial use and other purposes is therefore justified. The results of these analyses helped to increase the reputation of Slovenian scientists abroad, helped also to find foreign cooperation in scientific research and had a positive effect on publishing their results in international scientific journals. The way used to acquire the microanalyser could be an example of how an expensive analytical device can be acquired and used in small scientific communities.

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