AUTOMATION OF THE PROCESS OF OBTAINING HIGH-PURITY ZIRCONIUM FROM THE WASTES AND PRODUCTION RETURNS IN INDUSTRIAL FURNACES SKB-5025 AND APPARATUSES TS-40M

S.A. Lavrikov¹, M.L. Kotsar¹, A.O. Lapidus¹, S.G. Akhtonov², A.V. Aleksandrov², L.V. Ogorodnikov², A.A. Chernyshev², N.V. Kopysov² ¹OJSC "Leading Scientific Research Institute of Chemical Technology",

Moscow, Russia E-mail: kotsar@vniiht.ru; ²OJSC "Chepetsky Mechanical Plant", Glazov, Russi E-mail: post@chmz.net

A disadvantage of iodide refining of zirconium in industrial furnaces while reprocessing of wastes and production returns at the OJSC ChMZ is a low direct yield of metal into iodide rods and great energy consumption of the process. The aim of this work is to optimize the process by means of automated control. The paper deals with the creation of a test bench unit to automatize the iodide refining of zirconium in the OJSC ChMP. The main features of the test bench, the hardware and software of the automated unit as well as the results of its work during the operation are described. A scheme for the automation of 10 SKB-5025 furnaces by optimizing the total cost of computing equipment and software improvements was proposed and implemented in 2012.

The iodide refining method permits to obtain most pure zirconium in industrial conditions [1]. The highpurity zirconium obtained by the method of iodide refining is used as a component of furnace charge when smelting alloys for reactor purposes [2].

At present, the industrial processes of zirconium iodide refining in OJSC ChMZ are carried out in the apparatuses of the TS-40M type and in the SKB-5025 furnaces. The table below presents data on the chemical

composition of iodide zirconium obtained in these apparatuses [1]. The presented data make it possible to conclude that the actual content of total admixtures in zirconium rods is ~ 2.7 times less than the specified requirement value, while the content of individual admixtures, as a rule, corresponds to the limits of detection thereof in accordance with the techniques used.

Chemical composition and coeffi	icients of purification from	om admixtures in the	e process of zirconiu	m iodide refining			
in the TS-40 apparatus in the SKB-5025 furnace							

	Iodide refining of zirconium in the TS-40 apparatus			
Element	Chippings of sintered electrolytic powder bars	Iodide zirconium	C _{pur.}	Iodide zirconium, TU95.46-97
	Mass fraction of admixtures, %			Not more than
Nitrogen	0.0045	< 0.002	>2.2	0.005
Aluminum	0.0011	0.001	1	0.005
Beryllium	<0.003	0.0003		0.001
Boron	0.000042	< 0.00003	>1.4	0.00005
Hafnium	0.034	0.03	1	0.05
Iron	0.0081	0.004	2	0.03
Cadmium	-	0.00003	-	0.00005
Calcium	0.0036	0.00032	11	0.02
Oxygen	0.056	0.007	8	0.05
Silicon	0.004	0.002	2	0.008
Lithium	-	0.0001	-	0.0002
Manganese	0.0003	0.0003	1	0.001
Copper	0.0003	0.0003	1	0.003
Molybdenum	-	0.003	-	0.005
Nickel	0.003	0.004	0.75	0.01
Niobium	-	<0.006	-	0.01
Tin	-	0.0013	-	0.05
Lead	<0.003	0.003	-	0.005
Titanium	<0.003	0.002	-	0.005
Carbon	0,0046	0.003	1.3	0.008
Chromium	0,0032	< 0.003	1	0.02
Number of				
determined	16	21	-	21
admixtures				
Total admixtures	<0.1317	<0.0644	2.7	≤0.2863

The main disadvantage of this process is a low direct yield of metal, about 28%, into the iodide rod and great energy consumption. Besides, it should be noted that the process of formation of volatile zirconium compounds and their thermal dissociation over the hot wire in the TS-40M metal apparatus is not fully studied which does not allow to determine unambiguously the temperature mode in the course of the process to ensure the maximum velocity of zirconium deposition.

With the aim to optimize the process of zirconium iodide refining and to increase the yield of zirconium from one apparatus in the OJSC ChMZ, an automated test bench [5] was organized on the basis of one of the SKB 5025 furnaces with the following functions:

- to study a possibility of conducting an apparatus working cycle in an automatic mode without the attendance of personnel to exclude the human factor during the process;

- to develop optimum zirconium iodide refining control methods in the TS-40M apparatus in the SKB-5025 furnace;

- to adopt a decision on the duplication of this method on analogous furnaces of the shop sections, with the total number of which reaching several tens. The process of zirconium iodide refining has been used on an industrial scale for over 50 years and during this time there have often been made attempts to fully automate the process; nevertheless, the attempts have failed due to various reasons and have been limited to the development of local automation means.

From the point of view of the control system to control operation of the TS-40 apparatus in the SKB-5025 furnace, it is possible to point out two circuits dealing to a certain degree with the adjustment of temperature of the wire, the electric resistance of which is continuously reducing due to zirconium deposition and, hence, the increase of diameter of the rod [3, 4]:

- the control of the thyristor-controlled voltage regulator (TVR) as per the established volt-ampere characteristic;

- the control of dampers (gate valves) DU-150 and DU-500 and blower in the apparatus air cooling system by using the readings of the temperature sensors located in various parts of the SKB-5025 furnaces (Fig. 1).



Fig. 1. Apparatus TS-40M air cooling diagram in the SKB-5025 furnace of the test bench

The first circuit is based on the periodic comparison of the voltage values effective at the ends of the wire and the current passing through the wire, and the adjustment of voltage in accordance with the volt-ampere characteristic specified in the standard operating procedure as well as the corresponding stage changes of the transformer. The circuit is controlled by the shift operator with a frequency of once an hour by using the readings of the pointer instruments: a voltmeter and an ampere meter.

The second circuit envisages switching on and off of the blower and adjustment of angles of turn of the gate valves in the TS-40 apparatus air cooling system in the SKB-5025 furnace. It is also controlled by the shift operator with a frequency of once an hour in accordance with the standard operating procedure. Besides, local automation means are used on the basis of the KSP-3 devices for the registration of data and control of the apparatus air cooling system following the readings of separate thermocouples.

When selecting the hardware and software complex (HSC) of the automated test bench, a number of factors were taken into account and there was opted the CompactRIO equipment of the National Instruments

company, USA, and its graphical software LabView [6-8]. The factors that have been taken into account include a possibility of reconfiguring the measurement and control circuits of the system, user-friendly graphical medium for the development of the software with a great number of built-in functions and libraries for organization of data exchange and analysis designed to be used by engineers and researchers not quite well-versed in the field of software development. The long-

term experience of operating the LabView hardware and software environments in the OJSC ChMZ was also not the least significant factor.

The HSC for automation of the test bench has an architecture based on the use of a real-time controller for furnaces SKB-5025 and the concept of the reconfigurable input-output. Fig. 2 presents the block diagram of the HSC for one of the SKB-5025 furnaces.



Fig. 2. Generalized interface of HSC with sensors and actuating mechanisms for one of the SKB-5025 furnaces

The equipment from the NI is located in the cabinet in one compact crate. The sensors and the actuating mechanisms are connected directly or via the matching modules of the ADAM series of the Advantech company, a widely known supplier of equipment in the field of measuring and control systems (Fig. 3). The industrial computer of the Advantech company is located near the furnaces and the cabinet.

The analog voltage (~U) and current (~A) signals have a shape close to the sinusoidal form with the frequency of ~ 50 Hz which is a result of using the thyristor voltage regulator (TVR) for controlling the power on the wire of the TS-40M apparatus. Furnished to the analog input module are both the effective values of the voltage and current, and their instantaneous values to track the shape of the signals and to calculate the effective values thereupon in order to compare them with the input analogs. The period between the neighboring access samples used to track the signal shape is $84 \,\mu$ s. The minimum time between the measurements may be 0.2 s.



Fig. 3. The HSC equipment located in the cabinet of the test bench

The signals from the thermocouples represent slowly time-varying analog signals the measurement of which is performed with the same sampling frequency as the voltage and current, but with further averaging of the measured values. The angle-of-turn signals reflect the position of the DN-500 and DN-150 gate valves in the apparatus TS-40M air cooling system whose opening angle varies from 0 (the gate valve is closed) to 90 degrees (the gate valve is open). The control of the TVR is regulated by the analog signal which changes the phase angle of opening of the power thyristors of the TVR regulator. The signal to control the TVR is furnished when the measured voltage and current values deviate beyond the upper or lower boundary limit of the volt-ampere characteristic of the process of zirconium iodide refining.

The digital signal input module is used to read off the so-called «limit switches» of the actuating mechanisms of the gate valves used to signal about full opening or full closing of the gate valves, as well as to read out the emergency signal from the TVR.

To control the actuating mechanisms MEO of the gate valves, two relays for each mechanism are used as part of the digital signal output module. One relay is used for increasing the turn angle of the gate valve, the other relay is used for decreasing the angle. Other relay outputs of the digital signal output module are designed for selecting the transformer stage (1, 2 or 3), switching on (off) of the power supply of the TVR, heater and blower.

The computer is coupled with the real-time controller by serial interface Ethernet with the standard protocol TCP/IP. The controller is connected to the reconfigurable chassis through the inner bus - Internal PCI Bus. The chassis mounts the main component of the system - the FPGA micro circuit making it possible to adjust the functionality of all of the above-mentioned input-output modules.

The software of the test bench is developed for all three levels of the system and consists of the computer program (Host.vi), controller program (RT.vi) and that of the reconfigurable chassis (FPGA.vi) [6] with regard to the selected development environment of the LabVIEW software.

The controller and chassis programs ensure autonomous operation of the controller to collect data and issue controlling actions to the actuating mechanisms of the test bench and represent programs requiring least modification or correction. The control of the process in the apparatus and all the calculations are performed on the level of the computer of the higher system level.

The main functions of the computer program when carrying out the process are as follows:

- automatic control of voltage feed to the wire within the limits of the appointed volt-ampere characteristics;

- automatic adjustment of the positions of the gate valves in the TS-40M apparatus air cooling system in the SKB-5025 furnace;

- smooth shutdown and smooth feeding of voltage when changing the TVR transformer stages;

- computing true values of the system parameters by using the calibration coefficients;

- the response of the system to short-time current surges in the wire when the zirconium rods touch the apparatus casing walls;

- the development of the optimum temperature mode search algorithms for the apparatus air cooling system;

- the registration of data and events happening in the course of these processes in the files in the text format and in the Excel format;

- automation of the standard operating procedure for the conduct of the process in which operator actions are tied with the specific current intensity values on the zirconium wire making it possible to conduct the process without attendance of personnel by using the program that has been prepared beforehand.

Thus, the system functions only with the working computer which is quite permissible in case of a pilot test bench. The exchange of data with the real-time controller is performed by the use of network variables determined on the computer level. The structure of the program is presented in Fig. 4.



Fig. 4. Structure of the Host.vi. program

As seen from the figure, in addition to the main menu item «Process - Start», the program contains items for the execution of other auxiliary operations: tuning, calibration, testing and programming of operator actions when specific current strength values are reached on the zirconium wire, tuning of the allocation routes for the measurement results files, as well as for the events happening during the process. The operator interface (front panel as per the terminology of LabView) of the main menu item of the program for the conduct of the process in the TS-40M apparatus is presented in Fig. 5.

Fig. 6 presents a fragment of the computer program block diagram in the LabView development environment representing a graphical image of all the elements of the program operation algorithm. From the point of view of programming, this fragment consists of arithmetical, logical and other elements interconnected by conductors for the transfer of data. This makes the process of programming more obvious and more attractive for the researchers and developers for whom programming is only a portion of their work.

Starting since mid 2010 the test bench has been used both for the development of decisions to optimize the process modes of zirconium iodide refining in the TS-40M apparatus, and for the production of the finished product. The main criterion of the optimum conduct of the process has been greater yield of zirconium from this apparatus as compared with the average yield of zirconium from all other apparatuses. The following parameters have been subject to optimization: automatic regulation of voltage feed to the zirconium wire, changing the temperature mode in the apparatus cooling system in case of zirconium mass growth impairment on the wire, as well as methods of response of the system to current surges on the wire when the zirconium rods touch the apparatus walls, which results in the shutdown of the process or in the yield of subquality products.



Fig. 5. Operator interface for conduct of process on the test bench



Fig. 6. Fragment of the block diagram of the interface program

The results of the work of the test bench has shown a yield of iodide zirconium in the apparatus to exceed the average yield from other apparatuses by approximately 10%. Proportionally, the consumed power has been reduced and smooth drop and feed of electric power ensured when changing transformer stages. Besides, the amount of subquality products has been reduced due to completing the process of turning of zirconium rods up to the end, this process, if the rod touches the housing, causes short-circuiting and premature shutdown of the process.

The positive results obtained during experimental operation of the automated test bench in shop 60 of the OJSC ChMZ within one year of actually non-stop operation including more than two hundred processes were at the root of the proposals to swap the operation of the iodide refining section of shop 60 of OJSC ChMZ to the automatic control of the process.

It is apparent that simple copying of this automated test bench to a greater number of SKB-5025 furnaces (in the section of the shop there are several tens of SKB-5025 furnaces SKB-5025) is inexpedient both in terms of the total cost of the system, and in terms of arrangement of a great number of computers with monitors. Therefore, there has been adopted a decision to use only one computer (the master computer) to control the processes in all the furnaces, this computer being connected with the corresponding real-time controllers by the serial interface Ethernet with the standard protocol TCP/IP. Additionally, with the aim to increase the reliability and stability of operation of the whole system, each controller was equipped with a relatively inexpensive operator panel MT6070iH of the Weintek Labs, Inc [9] company with the screen size of 7".

Hence, when organizing a system consisting of 10 furnaces, there have been introduced changes into the structure of the HSC system, and into the distribution of the tasks and functions between the computer and the real-time controllers for each of the furnaces SKB-5025 permitting the latter to control the conduct of the process in the apparatus independently.

1. The hardware and software complex (HSC) of the pilot test bench, as the basis for the creation of the automated control system, is based on the NI CompactRIO. The composition and purpose of the NI CompactRIO modules for one furnace have remained unchanged, though the pattern of their organization in terms of the total cost of the system and arrangement of the equipment has assumed the view shown in Fig. 7. In this diagram the real-time controller NI cRIO-9073 with reconfigurable chassis 2M Gate FPGA and the input-output modules are shown in less detail as one unit; its composition and purpose of modules fully correspond to the HSC of the pilot test bench presented in Fig. 2.



Fig. 7. Generalized diagram of HSC interfacing of the automated control system of the zirconium iodide refining process using ten SKB-5025 furnaces

Each controller is equipped with an additional operator panel MT6070iH serving to start up and monitor the process directly near the furnace and to control the process (to perform final adjustment) at the given furnace in case of failure of the computer, or to carry out the process independently. The panel is connected to the controller via a serial port RS-232 being a part of the latter; the data exchange with the controller is further realized by using the Modbus [10] protocol.

The computer is coupled with the real-time controllers via the Ethernet serial interface with the standard protocol TCP/IP through a standard switch for the given number of channels.

2. The proposed system of automation of ten SKB-5025 furnaces presupposes updating of the software of the pilot test bench to adapt it with the HSC. For this purpose, the execution of the regulation algorithms has been transferred from the computer to the controller that allowed the process to be carried out without participation of the computer; and there has also been developed the panel operator software to control the process. Thus, there have been realized two control channels to control the process in the SKB-5025 furnace: from the computer and from the operator panel at the furnace.

Besides, there has been developed a data file structure for the computer with regard to the processes (logbooks of data and events happening in the course of the process) and a gamut of additional programs. They include the program of polling of the controllers to collect statistics data and to fill the logbooks and the program for the supervisor of the section allowing to monitor the course of the process simultaneously for all furnaces and, if necessary, to introduce corrections into the course of the process from his own computer.

Figure 8 presents the front panel of the supervisor at the computer to monitor the conduct of all the processes. It is shown that at this moment the processes are conducted on six SKB-5025 furnaces and the control is performed from the operator panels.

At present 10 SKB-5025 furnaces with automated control have been put into operation at the OJSC ChMZ. The process of connecting the furnaces is performed by the forces of the OJSC ChMZ without disrupting the general pace of operation of the shop sections. In 2013, 10 more furnaces are planned to be put into operation in accordance with the new technique.

The authors accord thanks to A.V. Andreev, V.A. Pogadaev, V.G. Moiseev, N.A. Yagovkin, N.G. Shavrin, V.S. Arkhangelsky for their participation in the development of the test bench and the execution of the experiments.



Fig. 8. The front panel of the supervisor on the computer to monitor the course of the process

REFERENCES

1. M.L. Kotsar, O.G. Morenko, M.G. Shtutsa, et al. Production of high-purity titanium, zirconium and hafnium by the method of iodide refining in industrial conditions // *Inorganic materials*. 2010, v. 46, №3. p. 332-340.

2. M.L. Kotsar, S.A. Lavrikov, V.I. Nikonov, et al. High-purity titanium, zirconium and hafnium in nuclear power engineering // *Nuclear energy*. 2011, v. 111, issue 2, p. 72-77.

3. V.G. Moiseev, O.G. Morenko, V.A. Pogadaev, et al. Apparatus for zirconium iodide refining - Patent

2261287 RF, IPC C22B34/14, C22B9/10. – Inventions. Useful models, 2005, №27, p. 377.

4. A.V. Aleksandrov, V.G. Moiseev, V.A. Pogadaev, et al. Method of control of the zirconium iodide refining process - Patent 2421530 RF, IPC C22B34/14, C22B9/00. – Inventions. Useful models, 2011, №17.

5. S.A. Lavrikov, M.L. Kotsar, S.G. Akhtonov, et al. Development of pilot test bench for automation of the process of zirconium iodide refining on the basis of the industrial furnace SKB-5025 // Non-ferrous metals. 2013, №2, p. 82-87.

6. E.D. Baran. *LabVIEW FPGA. Reconfigurable measuring and control systems.* Moscow: DMK Press, 2009.

7. J. Travis, J. Kring. *LabVIEW for all.* Moscow: DMK Press, 2010.

8. NI CompactRIO Control and Acquisition System. //http://www.ni.com/f/products/2/2177/ru/.

9. Graphical touch panel Weintek MT6070iH. Specification.// http:// www.weintek.com/global/Product/Product.aspx?Catego ry=Detail&Uid=MT6070iH&T=HMI

10. V.V. Denisenko. *Computer-aided control of production process, experiment, equipment.* Moscow: Hot line–Telecom, 2009.

Article received 30.08.2013

АВТОМАТИЗАЦИЯ ПРОЦЕССА ПОЛУЧЕНИЯ ВЫСОКОЧИСТОГО ЦИРКОНИЯ ИЗ ОТХОДОВ И ОБОРОТОВ ПРОИЗВОДСТВА В ПРОМЫШЛЕННЫХ ПЕЧАХ СКБ-5025 И АППАРАТАХ Ц-40М

С.А. Лавриков, М.Л. Коцарь, А.О. Лапидус, С.Г. Ахтонов, А.В. Александров, Л.В. Огородников, А.А. Чернышев, Н.В. Копысов

Недостатком процесса йодидного рафинирования циркония в промышленных печах при переработке отходов и оборотов производства в ОАО ЧМЗ является низкий прямой выход металла в йодидный пруток и большая энергоёмкость процесса. Целью данной работы является оптимизация процесса путём автоматизированного управления. Рассмотрены вопросы создания опытного стенда для автоматизации процесса йодидного рафинирования циркония в ОАО ЧМЗ. Описаны основные его функции, представлены аппаратная и программная части автоматизированного стенда, а также результаты его работы за время эксплуатации. Предложена и реализована в 2012 г. схема автоматизации работы 10 печей СКБ-5025 путём оптимизации общей стоимости вычислительного оборудования и доработки программного обеспечения.

АВТОМАТИЗАЦІЯ ПРОЦЕСУ ОТРИМАННЯ ВИСОКОЧИСТОГО ЦИРКОНІЮ З ВІДХОДІВ ТА ОБІГІВ ВИРОБНИЦТВА В ПРОМИСЛОВИХ ПЕЧАХ СКБ-5025 ТА АПАРАТАХ Ц-40М

С.А Лавриков, М.Л. Коцарь, А.О. Лапідус, С.Г. Ахтонов, А.В. Александров, Л.В. Огородніков, О.А. Чернишов, Н.В. Кописов

Недоліком процесу йодидного рафінування цирконію в промислових печах при переробці відходів і обігів виробництва у ВАТ ЧМЗ є низький прямий вихід металу в йодидний пруток і велика енергоємність процесу. Метою даної роботи є оптимізація процесу шляхом автоматизованого управління. Розглянуто питання створення дослідного стенду для автоматизації процесу йодидного рафінування цирконію у ВАТ ЧМЗ. Описані основні його функції, представлені апаратна і програмна частини автоматизованого стенду, а також результати його роботи за час експлуатації. Запропонована і реалізована в 2012 р. схема автоматизації роботи 10 печей СКБ-5025 шляхом оптимізації загальної вартості обчислювального обладнання та доопрацювання програмного забезпечення.