

## The 50-th anniversary of Scientific and Industrial Complex "Supermetal"

(A brief review of the scientific activity of Scientific and Industrial Complex "Supermetal" for 50 years)

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The history of Scientific and Industrial Complex "Supermetal" began in the early 1960s. At that time, there was set a task to increase the production of glass fibers, which were produced in aggressive environment, at high temperatures and mechanical loads. Only platinum and its alloys could withstand such operating conditions. For that period, the demand for platinum and palladium considerably exceeded the annual volume of their global production. The two-component Pt–Pd alloys, three-component Pt–Pd–Rh alloys, as well as more complex alloys Pt–Pd–Rh–Ir and Pt–Pd–Rh–Ru, were chosen for the research.

The alloys were tested in the conditions, similar to operating conditions of glass-melting apparatuses. The physical and chemical research of the alloys' compositions was carried out. The metallographic, electron and microscopic studies of the alloys' structures were performed, along with the choice of the modes of optimum melting and cold plastic working. On the basis of the obtained experimental data, the industrial alloys were developed for the glass-melting apparatuses. These alloys, introduced at the glass fiber plants, made it possible to save several tens of tons of platinum.

In the 1990s, the dispersion-strengthened platinum and its alloys were developed and industrially introduced. The non-affinage processing of rich platinum scrap and waste was developed and commercially implemented.

Another important line of business, launched in the XXI century, became the making of catalyst systems for the production of nitric acid and wire products for measuring devices. The medical products were also developed and introduced. Nowadays, more than a hundred Russian and foreign companies are the partners and customers of the Scientific and Industrial Complex "Supermetal".

**Key words:** platinum alloy, glass-melting apparatus, glass fiber, heat resistance, creep rate, sublimation, wastes, dense orifice field, sectional stamping, catalyst systems.

The history of Scientific and Industrial Complex "Supermetal" (SIC "Supermetal") began in 1962. The foundation of enterprise was started from the development of new, more economical alloys and materials, based on the platinum metals, which were used in the apparatuses for glass melting and glass fibers production\*. The usage necessity of platinum metals and alloys in glass-melting apparatuses is connected with the following operating conditions: 1,000–1,700 °C in air complex stress state, in contact with silicate melts, during the hundreds and thousands of hours.

The alloys for glass-melting apparatuses must have high exploitation characteristics at  $0.7–0.9 T_{\text{melting}}$ . At the same time, their cost and deficiency of components must be acceptable. On the basis of the fact, that the cost of palladium was 4–5 times lower than the cost of platinum and rhodium, the development of new alloys was started from the study of heat resistance and glass resistance of two-component Pt–Pd alloys in order to the replacement of the platinum alloy with 7% of rhodium. In those days, this alloy was used in apparatuses for glass-fiber production. However, as the structural materials, pure palladium and two-component Pt–Pd alloys were used only in individual parts of glass-melting apparatus. It was caused by the fact that certain units (parts) of glass-melting apparatus operated at the different temperatures and in the different stressed states.

In order to get an idea about the distribution of temperatures in individual zones of glass-melting apparatus, there

were made the direct measurements of temperature on the operating apparatus. There was obtained the distribution pattern of vertical and horizontal temperatures for 100–orifice and 200–orifice glass-melting apparatuses [1].

At the same time, the stressed state was calculated for the parts of glass-melting apparatuses, by example of the 400–orifice glass-melting apparatus of the 4-8/9 PIT (4-8/9ПИТ) type and the 4-13/12Shch195PIT (4-13/12Щ195ПИТ) type [2]. The mechanical stresses of the orifice plates under the influence of the glass melt pressure were determined, on the assumption of the loading diagram of a right-angled plate. This plate was hingedly supported with the edges and uniformly loaded, depending on the level of the glass melt over the orifice plate and specific weight of glass. The bending stress in the center of the orifice plate was calculated, which was equal to 2.9 MPa. It was recommended to use a supporting under-orifice cooler to reduce the bending stress of the orifice plate to 0.2 MPa and eliminate the deflection of the orifice plate. On the basis of the calculation data, there was chosen the optimum thickness of the orifice plate: 2–3 mm. Similar calculations of optimum thicknesses of the orifice plates were made to exclude the deflection in operation for other types of glass-melting apparatuses.

\* For the period since 1962 till 2003, all scientific developments were carried out and introduced with the participation and under the direction of Professor E. I. Rytvin (Doctor of Engineering Sciences, USSR State Prize Winner, Honored Metallurgist of Russian Federation)



Scientific Transactions of Professor E. I. Rytvin

The calculated stress of the side walls of the 4-8/9PIT type glass-melting apparatuses was 0.12 MPa with 1 mm of the side wall thickness. This stress was formed from the tensile stress, due to the weight of glass melt, and heat stresses, caused by the temperature difference in the wall thickness. According to the calculation data, the tensile stress for the 4-8/9PIT type glass-melting apparatus was 0.1176 MPa.

There was also held the stresses calculation in other parts of glass-melting apparatus. This calculation made it possible to determine the parameters of creep test for all two-component, three-component, and multicomponent platinum alloys. The creep tests and operation experience of glass-melting apparatuses made possible the approximate determination of the long-term strength:

$$\sigma_{10,000 \text{ hours}}^{1,400^\circ\text{C}} \approx 0.49 \text{ MPa} \text{ and } \sigma_{10,000 \text{ hours}}^{1,200^\circ\text{C}} \approx 1.96 \text{ MPa}$$

It was concluded that this stress level in individual units (parts) of glass-melting apparatus does not limit the service life of units for 10,000 hours.

The following researches made it possible to model the behavior of platinum alloys under the operating conditions of glass-melting apparatus:

- the researches of the creep of platinum alloys at 1,400 °C ( $\sigma = 4.9 \text{ MPa}$ ) in air and in alkali-free glass and alkali glass melts;
- the high-temperature tests at 1,700–1,800 °C in a melt of the high-modulus glass of VM (BM), VMP (BMП), UP (УП) type;
- the high-temperature tests in optical glass melts.

The creep rate of alloys of the Pt–Pd–Rh–Ru system was studied using the statistical methods of the experiments planning. The mathematical equations of the creep rate for the 1,300, 1,400 and 1,500 °C temperatures were obtained at the initial stress  $\sigma = 4.9 \text{ MPa}$ . The equations are true for any of two-, three-, or four-component alloys within the limits of alloying elements concentration of the considered system, where the exploration area of originally chosen alloys was restricted by the following contents: up to 30% (wt.) of palladium, up to 15% (wt.) of rhodium, and 10% (wt.) of ruthenium. The obtained models made it possible to determine the creep rate of any alloy in the part of the considered Pt–Pd–Rh–Ru system. The calculations made it possible to have the graphical dependences of the creep rate logarithm from

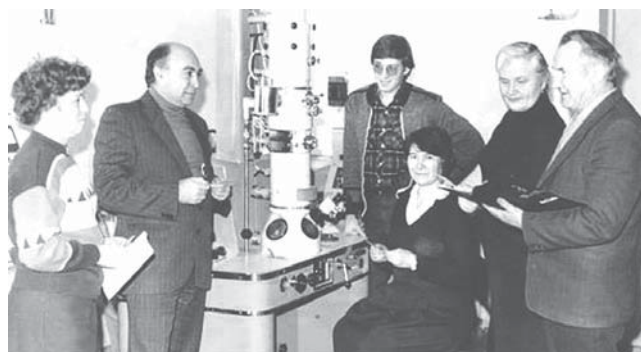
the composition of three-component Pt–Pd–Rh alloys and four-component Pt–Pd–Rh–Ru alloys. According to the statistic planning methods, there were obtained the experiments and mathematical equation models, for the description of the dependence of durability  $\tau$  (per hour), creep rate  $\varepsilon$  (% per minute) and unit elongation  $\delta$  (%) at 1,400 °C, and the initial stress  $\sigma = 4.9 \text{ MPa}$ , from the content of palladium and rhodium in a three-component platinum-based alloy [3].

The calculation data of the chosen three-component Pt–Rh–Pd alloys were confirmed by the experimental data at 1,400 °C and  $\sigma_{\text{initial}} = 4.9 \text{ MPa}$ . On the basis of the calculation data and the experimental data, the areas of optimum compositions were defined and industrial alloys for glass-melting apparatus were developed. At the same time, the research was carried out to determine the density of industrial alloys of platinum with 7% and 15% of the produced rhodium and palladium when melting in vacuum, argon and in air with different overheat levels, at different crystallization rates [4]. The influence of the prestrain and heat treatment modes on the structure and heat resistance of platinum alloys [5] was investigated. The recrystallization curves were created for the Pt–Rh alloy of the Pt–Pd–Rh–Ru system. There was studied the influence of impurities on the heat resistance of platinum alloys [6].

The electron microscope studies of the Pt–Pd–Rh–Ir system alloy (with 35% of palladium) made it possible to determine a certain connection between the level of deformation and subsequent annealing, and the dislocation structure and heat resistance of the alloy [7]. It was determined that the substructure, which is formed in the process of the cold plastic strain and subsequent annealing, is changed in the process of high-temperature creep. The optimization of the processing modes of platinum metal alloys made it possible



The collective of SIC "Supermetal" in 1972

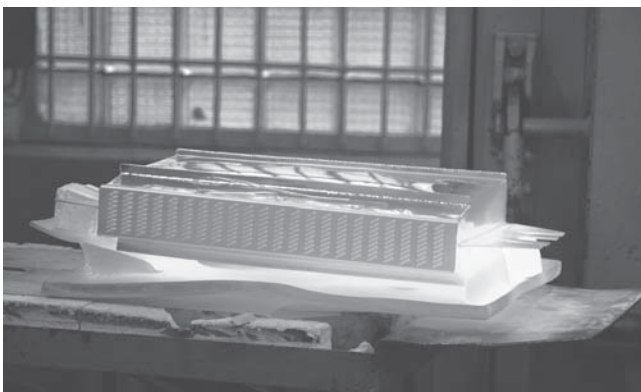


The winners of USSR State Prize: E. I. Rytvin (the second on the left), V. M. Kuzmin (the first on the right), L. P. Ulybysheva (sitting in the center). 1982



Melting of platinum metals

to increase considerably their life duration in glass-melting apparatuses, which had an influence on its service life. To the beginning of 1980s, there was approximately three times increasing of the service life of 400-orifice glass-melting apparatus (367 days instead of 100 days). It led to a certain increase in the loss of platinum metals during the operation, connected both with their air sublimation at 1,200–1,450 °C and with their dissolution in the glass melt. It was necessary to determine, which of the two factors is crucial. To answer this question, several tens of operating glass-melting apparatuses were taken under control. At the different operation stages of glass-melting apparatuses, the samples of glass fibers were taken, in which the content of platinum metals [8] was determined by neutron-activation analysis (in cooperation with the Institute of Physics of Latvian SSR). There was calculated the total weight of the platinum metals, which were dissolved in glass and to the glass fibers. It made it possible to determine the loss of platinum metals because of their air sublimation in the form of gaseous oxides  $\text{PtO}_2_{\text{gas}}$ ,  $\text{RhO}_2_{\text{gas}}$  or metallic gaseous palladium. Because of the dissolution of platinum metals in the glass melt, the loss of platinum metals and passing to the glass fibers is about one third of the total loss, and two thirds are due to the air sublimation of platinum metals. It was necessary to take into account the fact that the part of evaporated gaseous oxides  $\text{PtO}_2_{\text{gas}}$ ,  $\text{RhO}_2_{\text{gas}}$  or a metal gaseous palladium are condensed in the form of the reduced metal crystals on the refractory ceramics, with the



The 400-orifice glass-melting apparatus

following removing from it. This ceramics is a heat-shielding material for the glass-melting apparatus.

The researches made it possible to determine the specific loss of platinum metals (in grams) for 24 hours operation of each type of glass-melting apparatus or loss of platinum metals (in grams), referred to one ton of the produced glass fibers. The calculation procedure of the specific loss of platinum metals in operation was established in the 1970s–1980s. This procedure is still being successfully applied to determine the losses of platinum metals in operation of new modern types of glass-melting apparatuses and multi-orifice feeders, fabricated from new composite materials [9].

One of the directions in the problems of platinum economics is to reduce the specific consumption of the precious metal per mass unit of glass fibers, by the reduction of the dimensions (weight) of glass-melting apparatus. In the middle of 1970s, it was suggested to solve the problem by the reduction way of the orifice plates weight, making the orifices pattern more dense [10]. There was studied the development potential of solid-stamped orifice units of glass-melting apparatus with a dense arrangement of orifices. The 400-orifice plates of Pt–Rh–10 alloy and Pt–Rh–Au–5–4 alloy were chosen for the study. In order to the reduction of the stamping force, there was sometimes used the sectional stamping. One half of a plate (200 orifices) was stamped first, and then the other half was stamped. So it was experimentally proved that it was possible to fabricate the orifice units of glass-melting apparatus with dense pattern of gold-containing alloy orifices, using the method of cold die forging. For the first time, the idea of using a dense orifice field and sectional stamping of orifice unit of glass-melting apparatus, was tested in the middle of 1970s, and found its continuation in the 2010s in new small-sized glass-melting apparatus and multi-orifice feeders. Since 2002 till 2005, there were developed the typical multi-orifice designs of 800—, 1,200—, 1,600—, 2,400—, 3,200—, and 4,000— orifice feeders from Pt–Rh–10 alloy for the single-stage production of continuous glass fibers [11].

All designs were equipped with solid-stamped sections of orifice plates with a dense pattern of orifices, made of new materials.

The best heat- and glass-resisting platinum alloys were tested, and introduced at the glass fiber plants and, finally, saved some tens of tons of platinum to the end of 1980s. Thus,



Manufacturing division of the assembly of glass-melting apparatuses

the glass fiber industry's demand for platinoids was met, only using the internal capabilities of the country [12].

At the same time, two more scientific and technical problems of the state value were solved. According to this, the materials and equipment with an operating temperature up to 1,700–1,750 °C were developed on the basis of platinoids. This ensured the industrial production of extra-high-strength and extra-high-modulus glass fibers for the modern equipment. Laser and other special technologies were developed for the fabrication of very complicated types of platinum equipment for the production of polylayer optical fiber components, used in informational systems.

Thanks to the older generation of the specialists from SIC "Supermetal", the scientific and industrial potential of the company allowed the next generation of specialists to broaden their fields of activity in the 1980s–1990s. The dispersion-strengthened platinum and alloys were developed and industrially introduced [13]; the non-affinage processing of rich platinum scrap and waste [14, 15] was developed and commercially introduced. There was broadened the range of products and accessories for the high-quality special glass melting and growing of monocrystals and modern equipment for the production of continuous glass and basalt fibers [16].

Another important line of business at the SIC "Supermetal", launched in the XXI century, became the catalyst systems formation for nitric acid production; as well as the development and intensive industrial introduction of new materials for catalyst systems [17]; and the production of thermocouple wire and platinum wire for impedance transformers [18].

In conclusion, it is necessary to mention the development and practical application of a group of noble metal alloys for the dental prosthetics, which met the world standards [19, 20].

The foregoing brief information of the major directions of SIC "Supermetal" activity is the evidence of the company's wide scientific, technological, and industrial specialization. The achieved results and prospects of their practical usage in new and innovative developments give a hope for the bright future.

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