Glazunov’s electrography—the first electrochemical imaging and the first solid-state electroanalysis

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Abstract
Aleksandr Il’ich Glazunov developed the first electrochemical technique to image the surface of conducting solids giving the technique the name electrography. The electrographic images can mirror the distribution of elements on the surface of solid materials and also the electrochemical activity, caused by variations of “dissolution tension”. Thus, he has established for the first time a kind of spatially resolved electrochemistry. Electrography is also the first direct electroanalytical technique for solid materials. The present paper gives an account of his turbulent life in Russia, Czechoslovakia and Chile, and a discussion of his main scientific achievement, the development of electrography.

Keywords Electrography · Glazunov · Jirkovský · Imaging · Electroanalysis · Surface analysis · Solid samples

Aleksandr Il’ich Glazunov—his life as a Russian refugee

Aleksandr Il’ich Glazunov (Александр Ильич Глазунов) (Fig. 1) was born on January 20, 1888, in Saint Petersburg, Russia. His family (Fig. 2) has run a famous publishing house in this city [1]. The founder of this publishing house was Matvey Petrovich Glazunov (Матвей Петрович Глазунов) (1757–1830), the brother of the great-great-grandfather of Aleksandr Il’ich Glazunov. The Glazunovs published, besides law books and textbooks, the works of Pushkin, Lermontov, Zhukowskii, Turgenev, Goncharov, Ostrovskiy and many other famous Russian writers. Aleksandr Il’ich was the son of Il’ya Ivanovich Glazunov. Several members of the Glazunov family received the highest Russian awards and served as mayors of Saint Petersburg. Ivan Il’ich Glazunov, the grandfather of Aleksandr Il’ich Glazunov, was awarded the Order of St. Vladimir of the 3rd class with the assignment of the rights of hereditary nobility of the Russian Empire (Fig. 3). The famous Russian composer Aleksandr Konstantinovich Glazunov was the nephew of A.I. Glazunov’s grandfather.

In 1906, A.I. Glazunov graduated from the Imperial Business School with a gold medal and the title of candidate of commerce. Afterwards, he studied at the Polytechnic Institute, finishing the Faculty of Mining in 1912 with a diploma and the title of engineer of metallurgy and electrochemistry [4]. He then became assistant of the famous Russian chemist Nikolay Semënovich Kurnakov (Николай Семёнович Курнаков) (1860–1941) [5]. Kurnakov and Glazunov introduced the term “bertholoid” for compounds of variable stoichiometry at a meeting of the Russian Physico-chemical Society on May 10, 1912 [6]. From 1913 to 1917, he was the head of the Glazunov publishing house together with Mikhail Konstantinovich Glazunov (Михаил Константинович Глазунов) (1870–?) and the chemical plant on

The author dedicates this contribution to his dear friend and highly venerated colleague Professor Dr. George Inzelt, Eötvös Loránd University Budapest, on the occasion of his 75th birthday on November 2, 2021. I hope that this paper will find George’s interest as an electrochemist and science historian. This contribution is also an expression of my personal thanks to George for serving as Topical Editor of the Journal of Solid State Electrochemistry and ChemTexts — The Textbook Journal of Chemistry, and as co-editor of several books.

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Crimean peninsula and the Temryuk electrochemical factory. In 1918, he was elected as a member of the council for developing the Tavrida National University (Simferopol), the foundation of which was actively supported by V.I. Vernadsky. It is likely that Vernadsky proposed Glazunov as a member of that council. From a letter of Glazunov to Vernadsky, it is clear that they knew each other. In that letter from Kerch, Glazunov informed Vernadsky about the composition of gases, especially the H₂S content, evolving from a mud volcano near Temryuk (Taman peninsula, Krasnodar region) [7]. Later, Vernadsky met Glazunov in 1927 in Příbram [8]. A.I. Glazunov possessed a villa (Fig. 4) in Staryy Karantin, now part of Kerch on Crimea. This was one of the most beautiful villas of that town. Previously, the villa and the estate were sold to Aleksandr Il’ich Glazunov following the death of its previous owner, the counsellor Aleksey Petrovich Karpenko-Berezhetskiy. The estate had around 20.7 hectares of land. Aleksandr Il’ich Glazunov had a purely practical interest in the villa, as he could furnish it with equipment for physico-chemical studies of the mud ejected from the Bulganskiy mud volcanos (Fig. 5). This mud contains iodine, bromine and boron compounds. For the needs of the military department, Aleksandr Il’ich Glazunov donated in 1915 his physical and chemical laboratory (having around 90 m²) in that villa with all its equipment to the state [9], and he desired to continue his work there, aiming to explore the mineral resources of the Kerch peninsula. The villa was used as a headquarter of the Kerch District Revolutionary Committee and was ruined during WWII. Now, at its place a Lyceum of Arts is situated.

In 1919, following the departure of the German troops, red partisan detachments arrived in the Kerch district. The road from Stary Karantin to Kamys-Burun became the site of attacks on passers-by. At the end of March (according to the old-style calendar) of 1919, Aleksandr Il’ich Glazunov was captured by the partisans along with two officers on the road, when they were all riding together, apparently heading to his villa. Soon after, the officers were shot. The partisans, trying to establish friendly relations with the local intelligentsia, decided to keep Aleksandr Il’ich Glazunov alive. One of the partisans, Ivan Ovcharenko, writes in his memories of the affairs of his detachment: “He was treated very politely. They made him a bed of straw, fed him with eggs, milk, butter, in a word, made it clear in every possible way that no bandits gathered in the rocks ... but conscious revolutionaries ...”.

Glazunov was “offered” “to support the detachment financially”. Taking this opportunity, Glazunov wrote a note to his wife. Having received this unexpected message (probably, she did not longer hope to see him alive), she collected urgently—within a few hours—12 thousand roubles, borrowing it under any pretext in order to free her husband.

Here it needs to be mentioned that during the years 1917 to 1920, the political and military situation on the Crimea peninsula was chaotic and unstable due to the Russian Civil War. Many people left Crimea. A.I. Glazunov fled in 1921, first to Constantinople and soon after to Prague. The following information is from the Czech Biographic Dictionary [10] and the archive of Czech Technical University [4]. In 1922, Glazunov defended his doctorate at the Prague Technical University based on his thesis “Conductivity and hardness of zinc and cadmium alloys”, i.e. based on his research at Polytechnic Institute, Faculty of Mining, Saint Petersburg, in the group of N.S. Kurnakov. In 1922, he joined the Department of Metallurgy at the State College of Mining (Vysoká škola báňská) in Příbram as a substitute professor. In 1923 he was appointed associate professor and in 1931 full professor of physical chemistry, metallography and alloy theory, electrometallurgy and ironwork. He became the head of the new Institute of Theoretical Metallurgy, which he established.

1 Vysoká škola báňská literally translates as Mining High School, designating a kind of technical university. The most appropriate translation is State College of Mining; Mining Academy is another possible translation.
1935/1936, he was elected rector of the State College of Mining. For the period 1939 to 1945, i.e. when the German occupants closed all universities in Czechoslovakia, there is a
report in the Internet [11] and in a book [12] in which Aleksandr Il’ich Glazunov is mentioned as a consultant of the company Kalcium (Prague), which produced vitamins. Maybe his work there was only occasional because in [10] it is stated that A.I. Glazunov remained in Příbram during the German occupation and wrote books and papers. During the same period, the Kalcium company also employed Antonín A. Vlček (1927–1999), Rudolf Brdička (1906–1970) and, as apprentice, Rudolf Zahradník (1928–2020). Jaroslav Heyrovský (1890–1967) taught polarography to the people of the company.

After the liberation of Czechoslovakia in 1945, A.I. Glazunov started teaching again; and in 1946, after moving the school to Ostrava, he moved to that town in Northern Moravia. He was appointed a member of the editorial board of the newly established magazine Hutnické listy (Metallurgical Journal; https://www.hutnickelisty.cz/en/). He became an advisor to the Czechoslovak metallurgical industry, completed study trips abroad and renewed his active participation in international scientific meetings. He was an internationally respected expert, member of the Masaryk Academy of Labor (Masarykova akademie práce) and the Czechoslovak Chemical Society. In France, he was appointed a knight of the Order of the Legion of Honour (Légion d’honneur). He became a member of the international organizations Société de chimie industrielle, Institute of Metals, Iron and Steel Institute, Faraday Society, Société des ingénieurs civils de France and Association internationale pour l’essai des matériaux. He served on the editorial board of Métaux et Corrosion. According to his curriculum vitae [4], he had full command of Russian, French, English and German.

In 1946, he lectured in Paris on electroforming, and at the University of Geneva on the production and refining of aluminium. In January 1948, he went on a lecture tour to Switzerland. After returning to Czechoslovakia, and following the Communist February Coup in 1948, he left the country for
Chile. It is not surprising that he flew away from the communists, remembering his bad experience with the red partisans in 1919 on Crimea. He passed away in Santiago de Chile on October 21, 1951. This date is from the archive of the cemetery in Santiago de Chile (the date given in the Czech Biographic Dictionary is wrong). His grave still exists in the Cementerio General (Patio 83, bóveda 1258).

The scientific work of Aleksandr Il’ich Glazunov

It is not easy to find the publications of A.I. Glazunov since they appeared in badly accessible journals. The first noteworthy paper is certainly the above-mentioned publication with Kurnakov [6]. The problem of berthollides continued to interest Glazunov, as we can see from a paper published much later [13]. Despite developing electrography, which is presented in the next paragraph, A.I. Glazunov studied the use of electrochemistry in medicine, developing new methods of electrotherapy, dry iontophoresis and electro-intravenous injection. Together with Silvestr Prát (1895–1990), a renowned Czech plant physiologist, Glazunov proposed in 1937 [14] the method of electro-injection of metal cations in plant tissue. For this, a metal electrode (Fe, Ni, Cu, Ag, Au, Pb) was inserted into the tissue and by anodic oxidation the respective metal ions were released. The experiments were made to detect the location of the metal ions in the tissue and cells by microscopy following chemical reactions with reagents, e.g. hexacyanoferrate for iron ions and dimethylglyoxime for nickel. Another motivation of this work was to study the physiological action of the metal ions (in addition to the effect of the direct current).

Electrography

The main achievement of A.I. Glazunov is the development of electrography, a name that he coined for the first electrochemical imaging of solid surfaces. The term electrography has been used for a number of very different techniques. The Czech physicist Bartoloměj Navrátil (Sept. 2, 1848–April 12, 1927) was probably the first who used that term for the photography of corona discharge, later called Kirlian photography. Since that is completely unrelated to the “electrochemical electrography”, this will not be discussed here. Now, the term electrography is mostly used for recording electrophysiological activities, e.g. of the brain and heart, all that is also unrelated to the electrochemical technique. Electrographic printing is better known as xerography, whereas electrotyping for printing is also sometimes referred to as electrography.

Glazunov’s electrography is an electrochemical imaging technique for the surface of metals, alloys and ores, which possess electron conductivity. With that, he was for decades ahead of the later developments, i.e. scanning electrochemical microscopy and related techniques [15–19] and methods of local electrochemical analysis [20]. It can be also referred to as the first method for solid-state electroanalysis [21–23]. In early reviews [24], Helmut Fritz [25] has been equally credited for creating electrography. However, this is only partially correct, as Fritz did not describe a surface imaging technique, but an instrumental method to perform spot tests (similar to Feigl’s spot tests) by electrochemical oxidation of metals in contact with a moving wet paper stripe, soaked with a dissolved colour reagent. In later communications, Fritz described a number of variations, of what he called electro-spot-tests (“Elektro-Tüfelanalyse” in German) [26–33]. A.I. Glazunov introduced electrography on the 8th Congrès de Chimie Industrielle, Strasbourg, July 22–28, 1928, published in Comptes Rendus de 8e Congrès de Chimie Industrielle 1929 [34] (which I could not retrieve) and in [35]. In the Czech paper, he used already the term “otisk makrostruktury” which means “imprint of the macrostructure”. Figure 6 shows the two images of iron surfaces illustrating his paper [35].

The very instructive Fig. 7 showing the electrographic device was published by Rudolf Jirkovský (March 29, 1902, Pavlíkov–November 10, 1989, Ostrava). Jirkovský (Fig. 8) studied at Charles University Prague where he received a doctorate in 1927. In 1929, he joined the State College of Mining in Příbram, where A.I. Glazunov was already employed. Jirkovský worked from 1929 to 1931 on his habilitation, and from the literature, it is clear that he used and further developed electrography. Jirkovský worked during the German occupation at a gymnasium in Příbram, and after liberation of Czechoslovakia, he moved together with the State College of Mining from Příbram to Ostrava. There he soon became full professor and stayed as a highly recognized scientist who devoted his research and teaching to analytical chemistry, mainly for metallurgy (https://www.databazeknih.cz/zivotopis/rudolf-jirkovsky-20945). We owe him two reviews of electrography [24, 36], which greatly facilitate to understand the development of electrography. As Fig. 7 illustrates, the specimen to be studied is polarized in such a way that the surface is oxidized to produce soluble metal ions, which diffuse into the wet paper containing a reagent that gives a colour reaction with the metal ions. Hexacyanoferrate can be used for iron ions, and dimethylglyoxime for nickel ions. Fritz showed [29] that a surface dissolution of the sample can be accomplished by simply short-circuiting the aluminium base plate with the electrically conducting sample.

On April 22, 1937, A.I. Glazunov gave a talk at the meeting of the Österreichische Mikrochemische Gesellschaft (Austrian Microchemical Society), presumably in the Josephinum at
Währinger Straße 25, Vienna (Wien) [37], which was published in 1938 [38]. In this paper, he nicely outlined the features of electrography:

(i) It is capable of providing imprints of the distribution of elements in metals and conducting ores.

(ii) It can provide imprints exhibiting the distributions of domains having different “dissolution tension”, i.e. surface energies, may it be due to mechanical stress, crystallographic disorder or composition.

(iii) It is astonishing how A.I. Glazunov already in 1937 understood the electrochemistry of solid surfaces and thus the potential of electrography for surface characterisation: In [38], he wrote: “Both, different phases, as well as single crystallographic entities of the same phase, dissolve – according to their energy content – more or less easily under the influence of the electric current, which makes accessible the structure of the respective specimen. // The anodic dissolution of idiomorphic single crystals produces on their surface patterns, which can show us, how the tension is distributed, i.e., they can provide rather far-reaching information not only about the machining, but also about their formation. Further the distribution of atoms in the space lattice will be mirrored in the images, and thus we will be informed about possible deformations of the space lattice by machining.” (Here is the original German text: “Sowohl verschiedene Phasen, als auch die einzelnen kristallographischen Gebilde derselben Phase lösen sich in Abhängigkeit von ihrem Energiegehalt leichter oder schwerer unter dem Einfluß von elektrischem Strom auf, wodurch die Struktur des betreffenden Werkstückes entwickelt wird. // Bei anodischer Auflösung von idiomorphen Einkristallen entstehen auf der Oberfläche Figuren, die uns zeigen können, wie in ihnen die Spannungen verteilt sind, d. h., sie können uns nicht nur über die Bearbeitung, sondern auch über ihre Bildung ziemlich weitreichenden Aufschluß geben. Entschieden wird auch die Art der Verteilung der Atome im Raumgitter ihren Reflex auf diesen Bildern finden und gleichermassen erfahren wir, ob das Raumgitter an einigen Stellen durch mechanische Bearbeitung deformiert worden ist.”)

(iv) It is therefore correct to say that he established for the first time a kind of spatially resolved electrochemistry: the mapping of the distribution of “dissolution tension” or surface activity is not accessible by light microscopy, but it became accessible by his electrographic images.

(v) It can be used to assay the compactness and stability of metal coatings.

(vi) It can be used for quantitative determination of components of coatings;

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Fig. 6 Imprints of the surface of two iron specimens obtained by A.I. Glazunov using electrography [35]

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Fig. 7 Left side: Electrographic device: Al, the cathode (a metal plate); A, the anode (a metal indenter); N, the specimen; p, paper soaked with electrolyte and reagent; D and s, stand. Right side: Image of the nickel domains of a polished ore sample. The black areas are in reality red because of the nickel dimethylglyoxime complex. Reproduced from [24]
It can be used for quantitative determination of the thickness of coatings by a coulometric approach using current and/or voltage for the endpoint detection, while measuring the product of current and time (i.e. the charge for complete dissolution).

Finally, A.I. Glazunov wrote about the possibility to perform on-site electrographic analyses, e.g. in mines, with the help of a battery-driven device.

For all these applications, he gives examples and figures, which are unfortunately of too bad quality to be reprinted here. In that paper, A.I. Glazunov also refers to the Baumann probe [39]: That probe consists of pressing a sulphuric acid–soaked silver halide photographic paper on a plane iron surface so that the hydrogen sulphide evolved from sulphide inclusions produces a silver sulphide image depicting the distribution of the sulphide inclusions. This Baumann probe may have given him the idea to produce images by anodic oxidation of a metal surface with transfer of the produced ions to a reagent-soaked paper. In that publication, he explicitly states that it was Jirkovský who applied electrography for the first time to minerals and ores. Electrography has been also used in the USA by John H. Hruska (Hruška) (1897–1958) [40], a graduate of the State College of Mining in Příbram, who immigrated to the USA in 1923. It was also used in Great Britain [41, 42] and in other countries and became a rather popular technique in the post-war period. Figure 9 depicts a laboratory device, and Fig. 10 a pocket device, both fabricated by the London-based company Baird and Tatlock Ltd., to perform electrographic tests. That company operated from 1897 to 2012. Whereas the laboratory device was suitable for imaging electrography, the pocket device was intended to perform only spot tests on a certain surface location. The surface was oxidized, and the ions sampled on a piece of paper. On that paper, qualitative metal detection could then be performed with reagents, as it is the principle of Feigl’s spot tests. Since 1918, Fritz Feigl (1891, Wien, Austria–1971, Rio de Janeiro, Brazil) worked on spot tests, i.e. putting a drop of an analyte solution on paper and adding another drop of a reagent solution, as to produce a specific colour for identifying the analyte [43, 44].

Figure 11 depicts electrographic arrangements for local microanalysis of small surface areas [45]. Clearly, the use of an electrolyte-filled capillary can be regarded as a very early forerunner of modern capillary cells, as developed independently by Böhni [46, 47] and Lohrengei [48]. It is also worth mentioning that Glazunov published already in 1932/1933 a thin-layer electrochemical cell to study the rate of electrocrystallisation of copper [49] and of silver peroxyde in 1934/1936 [50], i.e. about 30 years before F.C. Anson started his work on thin-layer electrochemistry [51]. The nucleation and growth of crystals were a question, which he did not only address in case of electrodeposition, but more generally also for crystallisation of liquids [52, 53]. Also, in the later cases, he used a thin-layer cell for microscopic observation of the crystallisation rate.

The breadth of Glazunov’s interests and expertise in electrochemistry is also evident from a patent that he filed in 1948 [54]. In this patent, Glazunov suggests a three-layer electrolysis, with the primary produced aluminium sandwiched between two molten electrolyte layers. The primarily produced aluminium is acting as a bipolar electrode with the upper layer of aluminium being the cathode of primary electrolysis, and the lower surface of that aluminium layer being the anode. The anodically dissolved aluminium is reduced back to high-purity aluminium on a molten aluminium layer, which is separated from the primary aluminium layer by vertical walls and floating on top of the lower electrolyte layer. Although the patent was obviously never used because the aluminium produced in modern electrolysis processes has already a sufficient purity for commercial use, it is certainly a genial proposal and it may still be realized in one or another way in the future.2

2 Private communication of Andrey Yasinskiy
Conclusions

Glazunov’s scientific interests covered a very wide field. Despite his military-centred and analytical work between 1915 and 1921, the following topics have to be mentioned: He was concerned to answer the question what a chemical compound is. From the times of research with Kurnakov, nonstoichiometric compounds, solid solutions and their structure remained a focus point of his activities, which he transferred to Příbram in Czechoslovakia. Attempting to understand the structure of alloys, he developed electrography. His interest in the structure of solids led him to study the kinetics of electrocrystallisation, and generally of crystallisation. For this, he developed thin-layer cells allowing microscopic measurements of the crystallisation rate. A complete presentation of his scientific research is beyond the scope of this paper and has to be postponed for future.

Here follow some conclusions, which can be drawn from Glazunov’s life:

(i) Even though we do not exactly know what was the chain of thoughts that inspired A.I. Glazunov to create electrography, it is likely that he was methodologically inspired by the Baumann probe [39] (see before) and scientifically driven by his interest in the structure of solids. If so, it is a good example for the success of transferring ideas from one topic to another, in order to solve a scientific problem.

(ii) Despite the fact that A.I. Glazunov announced electrography on large international conferences, he did not publish his research in widely distributed international journals, but rather specialised and local journals. His major disciple, Rudolf Jirkovský, spread the message in 1934 by writing a review in German (at that time worldwide understood by scientists) in a somewhat wider circulated journal [24]; however, this was a bit too early, as the developments of the next two decades could not be included. Certainly, the turmoil of the Second World War and its aftermath also impaired the international recognition of electrography. Much later, in 1951, H.W. Hermance and H.V. Wadlow from the Bell Telephone Laboratories have given a very detailed review on electrography.

Fig. 9 Laboratory electrograph of the company Baird and Tatlock (London) Ltd.: Left side: A, voltage control (0–10 V); B, voltmeter; C, range switch; D, ammeter; E, timer; F, light for “power on, timing motor running”; G, light for “output on”. Right side: A, aluminium counter electrode; B, pressure bar; C, hand wheel to vary the pressure; D, scale to show the thrust. Reproduced from Ref. [41] with permission from The Royal Society of Chemistry.

Fig. 10 Electrographic device for local microanalysis of small surface areas: A, body to hold two batteries; B, battery; C, slotted contact ring; D, aluminium plunger, which is spring loaded, and connects to the negative pole of the battery via a simple switch. Reproduced from Ref. [41] with permission from The Royal Society of Chemistry.
[55], which clearly shows the interest of industrial laboratories in electrophotography for fast analyses.

(iii) From today’s perspective, it is surprising that we could not find indications of intimate contacts between Glazunov and Jaroslav Heyrovský’s group in Prague, although Glazunov probably met Heyrovský. Such a cooperation, or at least an exchange of ideas, could have been very beneficial for both electrophotography and polarography.

(iv) The tragic fate of A.I. Glazunov is an example of how creative and determined people go their way—provided they have the financial means—despite setbacks caused by political interferences. Another lesson from his life, hopefully received by politicians, is as follows: give intellectuals all freedom to develop their faculties. Do not restrict and control them by political doctrines.

(v) Why should we remember Glazunov’s electrophotography? However interesting this may be for the historian, we may possibly benefit from the idea when faced with modern problems of solid-state analysis, which conquers more and more remote fields of applications, e.g. archaeometry and metal dating [56–58]. It is good to see that at least in Czech Republic, at the University of Pardubice, electrophotography is still included in the curriculum [45]. Although the interest in electrophotography slowly expired in the 1950s [59–62], there is still one paper from 1977 [63] reporting a (not completely satisfactory) trial to use electrophotography for quantitative analysis. I hope that the present paper may spark the phantasy of scientists and lead to new ideas.

(vi) Finally, Glazunov’s oeuvre shows how important it is that a scientist has a wide range of interests so that he is able to inseminate various fields of research.

This paper is meant to supplement the book Electrochemistry in a divided world [64], which is devoted to the fate and achievements of Eastern European electrochemists during the era of political division of the world in the twentieth century. It is a continuation of the author’s attempts to recognise Russian electrochemists [65, 66] who have made important contributions to science but are still less known in the Western world.

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