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STRUCTURAL CHARACTERIZATION OF PLATINUM FOIL FOR NEURAL STIMULATING ELECTRODES USING A FOUR-POINT RESISTIVITY-MEASURING DEVICE

STRUKTURNE LASTNOSTI PLATINSKE FOLIJE ZA ELEKTRODE PRI STIMULACIJI ŽIVCEV, UGOTOVLJENE Z NAPRAVO ZA ŠTIRITOČKOVNO MERJENJE UPORNOSTI

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In the past few decades, considerable efforts have been devoted to develop neuroprosthetics that interface selectively with the human nervous system via multi-electrode spiral cuffs, using implantable electronic devices. The objective of this study was to investigate the structural properties of a cold-rolled platinum foil used to manufacture multi-electrode spiral nerve cuffs. To achieve this objective, thick cold-rolled platinum foil strips 0.03 mm with 99.99 % purity were used. For this purpose, the strips were mounted into the sample holder within the furnace of a custom-designed set-up. The resistivity measurements were made using a 4-point probe technique in which the strips were subjected to dynamic annealing in an argon atmosphere within the temperature range between room temperature and 900 °C. Finally, the microstructures of the strips, prepared using standard metallographic techniques, were investigated using light microscopy. In the resistivity measurements, a small change is observed at ≈280 °C. This change could be explained as the partial recovery elicited by a decrease of the dislocation density. Above 500 °C, a significant decrease in the resistivity was recorded, and the decrease reached a maximum at ≈750 °C. These results provide a deeper insight into the fabrication of platinum foil to be used in the further development of multi-electrode neural stimulating spiral cuffs. The most important finding is that the results of the resistivity measurements provide one criterion for selecting materials, and that the appropriate thermal and mechanical working processes are required to fabricate stimulating electrodes. These results may make cold-rolled platinum ribbon the best material for the long-term application of multi-electrode spiral cuffs in SNS.

Keywords: selective nerve stimulation (SNS), platinum electrodes, annealing, recrystallization, microstructure, resistivity, light microscopy

V zadnjih nekaj desetletjih je bilo pri razvoju elektronskih živčnih protez, ki prihajajo preko večelektrodnih spiralnih objemk selektivno v stik z živčnim sistemom človeka, namenjenih veliko naporov. Cilj raziskave je bil preiskati strukturne lastnosti hladno valjane platinske folije za izdelavo večelektrodnih živčnih spiralnih objemk. Za dosego tega cilja so bili uporabljeni trakci iz 0,03 mm debele hladno valjane platinske folije čistosti 99,99 %. V ta namen so bili trakci montirani v nosilec vzorcev v peči posebej izdelanega merilnega sistema. Meritve upornosti so bile izvedene z uporabo 4-točkovne merilne tehnike, pri kateri so bili trakci dinamično žarjeni v argonu v temperaturnem območju med sobno tempreraturo in 900 °C. Na koncu je bila mikrostruktura trakcev, pripravljenih po standardnih metalografskih tehnikah, preiskana z uporabo svetlobne mikroskopije. Pri meritvah upornosti je bilo opaziti majhne spremembe pri temperaturi približno 280 °C. Te spremembe je mogoče pripisati delnemu okrevanju, ki ga povzroči zmanjšanje gostote dislokacij. Pri temperaturah nad 500 °C pa je bilo opaziti znaten padec upornosti, ki je dosegel največjo vrednost pri temperaturi približno 750 °C. Dobljeni rezultati omogočajo poglobljen vpogled v izdelavo platinske folije, uporabne pri nadaljnjem razvoju večelektrodnih stimulacijskih spiralnih objemk. Najpomembnejši sklep je, da rezultati meritev upornosti podajajo enega od meril za izbiro materiala ter primernih procesov mehanske predelave ter toplotne obdelave folije za izdelavo stimulacijskih elektrod. Na osnovi tovrstnih rezultatov lahko preverimo, ali je ustrezno hladno valjana platinska folija najboljši material za dolgotrajno uporabo pri večelektrodnih spiralnih objemkah za selektivno stimulacijo živcev.

Ključne besede: selektivna stimulacija živca, platinske elektrode, žarjenje, rekristalizacija, mikrostruktura, upornost, svetlobna mikroskopija

1 INTRODUCTION

In the past few decades, considerable scientific and technological efforts have been devoted to developing neuroprostheses that interface the human nervous system with implantable electronic devices. Potential applications include limb prostheses, bladder prostheses, cochlear and brain-stem auditory prostheses, retinal and cortical visual prostheses, cortical recording for the cognitive control of assistive devices, vagus nerve stimulation and deep brain stimulation for essential tremor, Parkinson's disease, epilepsy, dystonia, and depression. However, all the applications require electrodes characterized by high spatial and nerve fibre type selectivity and low impedance for recording and safe reversible charge injection for stimulation at the same time. With this in mind, an understanding of the electrochemical mechanisms underlying the behaviour of neural stimulation and recording electrodes is important for the development of chronically implanted devices, particularly those employing large numbers of electrodes.[1,2]

Since the installation of such a multi-electrode system onto a peripheral nerve also causes mechanical issues, the designer must consider and optimize both the aforementioned issues. To minimize the mechanical issues such as abrasive or compressive injuries and fitness difficulties in the application of multi-electrode systems, flexible substrates and appropriate metal materials have emerged.

In the area of Functional Electrical Stimulation, multi-electrode cuffs have been used in peripheral nerves for stimulation as well as for the recording of an electroneurogram for more than 35 years.[3–5]

In the last two decades, particular attention is being paid to vagus nerve stimulation, techniques that are to be used as a method to treat a number of autonomous nervous-system disorders.

In this regard, selective nerve stimulation (SNS) is usually delivered from a group of three electrodes (triplet) within the spiral cuff installed onto the nerve. The efficacy of SNS is dependent exclusively on localizing charge delivery to specific populations of nerve fibres. Charge delivery, however, is influenced by the electrode-tissue interface, where a transduction of charge carriers from electrons in the metal electrode to ions in the tissue occurs.

Electrodes for SNS face electrochemically harsh working conditions. At the same time, it is imperative that in contact with the nerve tissue, the electrodes must remain non-toxic and non-reactive with the tissue. That means while injecting a required amount of charge, the formation of any harmful reactions should be avoided.[6–8]

For SNS, different materials that support charge injection by capacitive and faradaic mechanisms are available. These materials have certain advantages and limitations. Among the criteria that must be considered when choosing the material for electrodes that make electrical contact with a neural tissue are the mechanical characteristics of the material.[9]

The noble metal commonly used as a stimulating electrode material which is capable of supplying adequate electrical charge to activate neural tissue is pure platinum.[10] As with most metals with a face-centred cubic structure, platinum is a very ductile metal.[11] High-purity platinum is non-toxic, insoluble in mineral and organic acids and does not corrode or tarnish. However, soluble salts are highly toxic by deposition within the internal organ tissues.

Apart from the chemical inertness, platinum has a number of physical properties of great value for its use in the technology of implantable stimulating electrodes.[12–18] These include general properties, mechanical properties and physical properties:

General Properties

Density: 21.45 g/cm^3
Thermal Conductivity: 0.716 W/(cm K) at 298.2 K
Electrical Resistivity: 105 nΩ m at 293.2 K

Mechanical Properties

Young's modulus: 168000 MPa
Tensile Strength: 125–165 MPa (annealed at 426.8 K)
Elongation: 30–40 % in 50 mm (annealed at 426.8 K)
Tensile Strength: 205–240 MPa (hard drawn, 50 % cold worked)
Elongation: 1–3 % in 50 mm (hard drawn, 50 % cold worked)
Elastic Modulus: 171000 MPa (annealed at 426.8 K) at 293.2 K
Elastic Modulus: 156000 MPa (hard drawn, 50 % cold worked) at 293.2 K
Yield strength: 38-180 MPa
Vickers hardness: 549 MPa

Physical Properties

Absolute electrode potential: 4.44 ± 0.02 V at 298.2 K
Melting temperature: 1495.1 K
Thermal expansion: 8.8 $\mu m \cdot m^{-1} \cdot K^{-1}$ at 298.2 K

In the electrical stimulation of nerves, platinum is often used in the form of the pure metal. Namely, impurities and alloying elements may adversely affect both its mechanical characteristics and its stability against corrosion in physiological media. This metal injects charge by both faradaic reactions and double-layer charging. The relative contribution of each process depends on the current density and the pulse width, although the faradaic processes predominate under most neural stimulation conditions.[10,19] However, in a SNS with a high charge density, pH shifts causing irreversible changes in tissue proteins, metallic dissolution products, gross hydrogen and oxygen gas bubbles, and oxidized organic and inorganic species, could occur.[20] It is of a crucial importance that the electrodes in SNS function without facing degradation over a prolonged time period.

Although the charge-injection limits of Pt are based on avoiding the electrolysis of water, Pt dissolution can occur at lower charge densities.[21]

The principal approach to controlling the interface voltage in SNS has been the use of charge-balanced biphasic stimuli, having cathodic and anodic phases that contain equal but opposite charge. However, even with charge-balanced stimulating pulses it is possible that the interface voltage may reach levels where electrochemical reactions can occur.[22,23]

Mechanical fitness is another important issue in multi-electrode systems design. Low strength, typical of a platinum of high purity (99.93 %), is accepted in stimulating and recording electrodes, despite being a significant disadvantage. To optimise the parameters for the thermal and mechanical processing of platinum a knowledge of its rheological characteristics, including deformation resistance, is required.[24–26] The deformation resistance, however, is considered in terms of the uniaxial compression or tension of the sample under conditions of plastic deformation, resulting in lattice defects. With this regard, the cold strength depends on the degree to which the object has been deformed: the more lattice defects the harder the metal. Grain boundaries disrupt the motion of dislocations through a material, so reducing crystallite size is a common way to improve the strength. It was assumed that during cold deformation, the deformation resistance depends only on the geometric parameters of the change in shape. When a metal is annealed, however, it loses this strength via the reaction known as recrystallization. The annealing temperature range for 99.93 % platinum is 400–1000 °C, and depends on the degree of cold-working.[27–29]

Resistivity, sometimes called the Specific Resistance, is one of the fundamental electrical properties of a particular material which gives it electrical resistance.[30] Resistance can be calculated by using a knowledge of both the resistivity of the material of a particular object and the shape and geometry of the object. Accordingly, the electric resistance of a square sheet is independent of the size of the square, but depends only on the sheet resistivity and the thickness of the sheet.

Characterizing the resistivity of a material for the SNS electrode could help to determine the long-term charge delivery to the nerve without harmful effects for both the neural tissue and the electrode.

It has been common knowledge that, in general, the resistivity of a metal is higher when cold-worked than in an annealed state.[31,32] Namely, grain boundaries are defects in the crystal structure and they tend to decrease the electrical conductivity of the material. It was found that platinum of the highest purity almost completely recovers its electrical properties at a temperature as low as 300 °C, although a slight further recovery occurs with an increase of the annealing temperature up to 1450 °C.[33] The result of annealing is the diffusion of dislocations and their cancellation through encounters with dislocations of the opposite sign. In this regard, the increase in hardness and resistivity due to the strain condition obtained during cold-rolling could be reduced to zero by annealing within the temperature range between 400 °C and 800 °C.

Improved, implantable, multi-electrode stimulating systems with greater strength, desirable for an individual electrode, may be achieved through plastic deformation during cold-rolling and partial annealing by carefully choosing the appropriate annealing regimes.

In order to set up the appropriate working cycles, metallographic analysis revealing the microstructure of platinum after each cycle should be used. The most convenient way to reveal the microstructure of cold-rolled and/or thermally treated platinum foil used for stimulating electrodes is by electrolytic etching.[34,35] Namely, work hardening and thermal treatments cause significant changes to the microstructure of the platinum with respect to the as-cast condition.

The objective of this study was to investigate the temperature-dependent structural properties of a cold-rolled platinum foil used to manufacture stimulating electrodes within the spiral nerve cuffs for SNS using a custom-designed four-point resistivity-measuring device.

2 METHODS

The foil was obtained by means of cold-rolling of the sheet material. In the most labour-intensive method of foil preparation, a small piece of the ingot (chemical composition of the ingot: Pt 99.99 %, Rh 0.01 %) was extracted and, after initial working cycles, the material was annealed and subsequently rolled flat on a mill to obtain a thick foil 0.15 mm. Because platinum has a very high stiffness, work hardening during working cycles made the foil appear excessively hard. In addition, the platinum foil experienced severe local hardening arising from the high strain rates during cold-rolling. We presumed that a gradual increase in the dislocation density

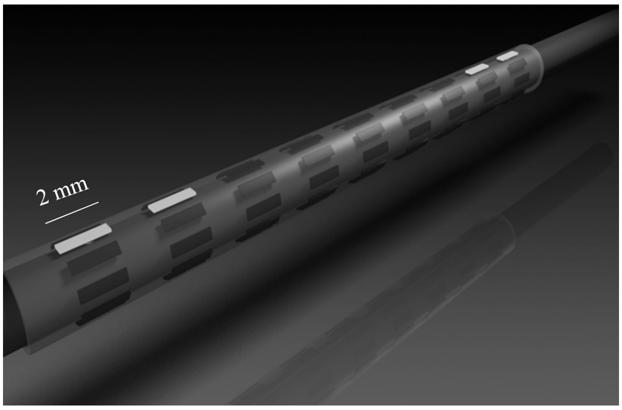

Figure 1: The 99-electrode spiral cuff and an arbitrarily chosen longitudinal row of electrodes

Slika 1: Prikaz 99-elektrodne spiralne manšete in poljubno izbrana vzdolžna vrsta elektrod occurred, causing increased hardness and a localized loss of ductility. To reduce the dislocation density, renew the softness and ductility, and provide a correct orientation of the metal grains for further cold rolling, recrystallization was promoted by subsequent annealing at 700–800 °C. Finally, the foil was rolled to achieve the final thickness of 0.03 mm on a mill with diameter rollers 300 mm, using varying degrees of cold-working.

The long-term electrochemical stability of platinum stimulating electrodes within the multi-electrode spiral nerve cuff, shown schematically in **Figure 1**, may be connected with the resistivity of the grain boundaries and its change with time.

We measured the electrical resistivity of the platinum strips currently used to manufacture the electrodes used in the 99-electrode spiral nerve cuff. For this purpose, test strips were cut from the foil, with their long axes oriented along the rolling direction. The resistivity measurements were made with a modified 4-probe (also called Kelvin probe) resistance-measurement technique.

For this purpose, a high-temperature resistivity measurement device shown schematically in **Figure 2**, designed by a research team at the Department of Materials and Metallurgy, Faculty of Natural Sciences and Engineering, University of Ljubljana and Marko Pribošek, an individual investigator, was used.

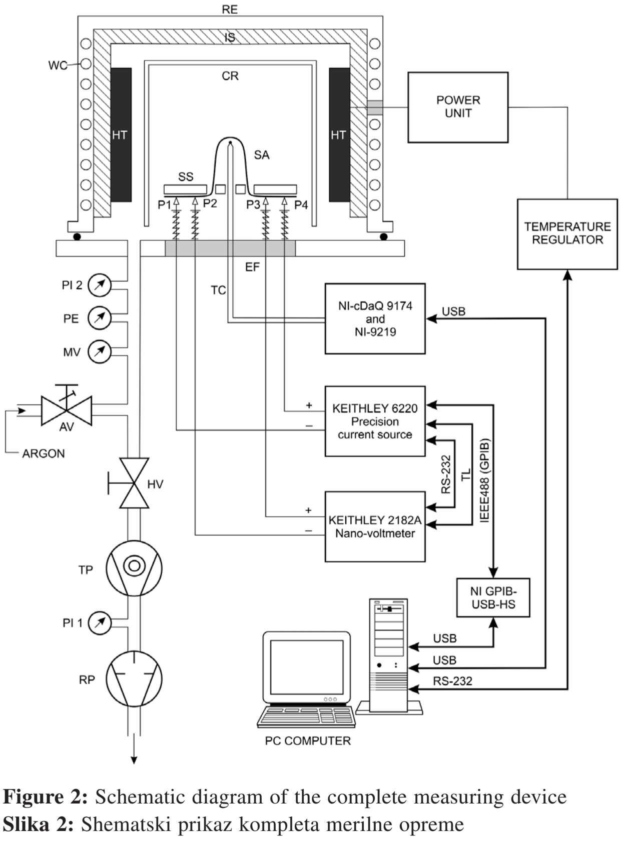

Figure 2: Schematic diagram of the complete measuring device
Slika 2: Shematski prikaz kompleta merilne opreme

Legend to the figure: RE- Recipient, IS- Isolation, CR- Ceramic retort, WC-Water cooling, HT- Heater, SA- Sample, SS- Sample support, P1, P2, P3, P4-Spring-loaded measuring points or contacts, EF- Electric (cable) feed through, TC- Thermocouple, RP- Rotary pump, TP- Turbomolecular pump, HV- High-vacuum valve, AV- Argon dosing valve, PI 1- Pirani vacuum gauge, PI 2- Pirani vacuum gauge, PE- Penning vacuum gauge, MV- Membrane vacuum gauge

The main parts of the measurement device are a custom-designed furnace that could withstand a temperature up to 900 °C and the sample holder. To maintain a reliable contact at the sample holder during the measurement, the strips were contacted by four spring-loaded platinum probes.

Direct current was supplied via two probes (source and sink) that were connected to the current source (Model: 6220, Keithley Instruments, Inc., Cleveland, Ohio, USA), and the voltage was measured between the other two probes, connected to the nanovoltmeter (Model: 2182A, Keithley, Keithley Instruments, Inc., Cleveland, Ohio, USA[36]; Model 6220 DC Current Source and Model 6221 AC and DC Current Source[37]; Model 2182 and 2182A Nanovoltmeter[38]).

The complete measuring device and the sample holder housing four spring-loaded platinum probes are shown in **Figure 3**.

To maintain a linear pattern of heating, the system included a digital proportional-integral-derivative (PID) controller, which uses the principle of a "continuous" furnace controller. The PID controller was interfaced with a computer via an RS232 port. A thermocouple (K-type) was used to measure the furnace temperature, while a Pt-100 sensor was used to measure the sample temperature. The data on temperature were retrieved using a USB data-acquisition module (Model: NI-9219, National Instruments, Corporation, Austin, Texas, U.S.A.) and a USB data-acquisition chassis (Model: NI cDAQ-9174, National Instruments Corporation, Austin, Texas, U.S.A.). With this configuration, the test current was forced through the strip resistance using one set of test leads, while the voltage across the sample was measured using a second set of leads, called the sense leads. Although some small current may flow through

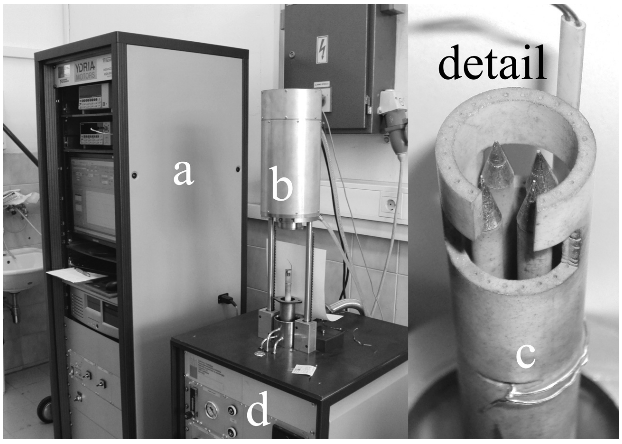

Figure 3: Complete measuring device: a) measuring and computer unit, b) furnace, c) four spring-loaded platinum probes (detail), d) vacuum unit
Slika 3: Celotna merilna naprava: a) merilna in računalniška enota, b) peč, c) platinasta sonda s štirimi vzmetmi (detajl), d) vakuumska enota

the sense leads, it is usually negligible (typically pA or less) and can generally be ignored for all practical purposes. Because the voltage drop across the sense leads was negligible, the voltage measured by the nanovoltmeter was considered to be essentially the same as the voltage across the resistance. Both instruments worked in tandem; the intercommunication was realized via a Trigger Link (TL) and an RS232 serial port interfaced with the computer using a data bus IEE488 (GPIB), an interface (Model: NI-GPIB-USB-HS, National Instruments Corporation, Austin, Texas, USA), and Lab-view software (Version 10.0, National Instruments, National Instruments Corporation, Austin, Texas, USA).

Platinum strips were subjected to annealing and, at the same time, the electrical resistance of the grain boundaries was measured using a direct method.[30] For the measuring conditions, the strips underwent annealing in an argon atmosphere using a linearly increasing temperature range from room temperature to approximately 900 °C, so as to achieve recrystallization. The heating rate was 5 K/min. This temperature range was initially estimated to be high enough to ensure complete recrystallization.[24] Under these conditions the temperature distribution during annealing was uniform along the strip in the argon atmosphere. In the measurements, the strips were so placed that the grain boundaries were oriented predominantly parallel to the length of the strip. Afterwards, at arbitrarily chosen points on the strip, platinum probes connected to the potential leads were mechanically pressed to the strip. An electrical measuring current of approximately 2–3 A/mm^2 was then passed through the strip and a potential difference of approximately 10 μV was measured using the aforementioned nanovoltmeter, having an input resistance of 50 MΩ.

To reveal the changes in the microstructure that occurred after the cold-roll hardening and the recrystallization thermal treatments of the strips, optical metallography was employed. To obtain this goal, the most convenient way, namely electrolytic etching, was used.[34,35] The preparation of the metallographic specimens consisted of the following four steps: sectioning the platinum foil into the strips, embedding the strips into the epoxy resin, mechanical grinding and polishing of the specimens, electro-polishing of the exposed metallographic section in a controlled-atmosphere glove box, and electrochemical etching for microstructure detection.

For the electrolytic etching, the polished specimens were immersed into the most widely used electrolytic solution, i.e., a saturated solution of sodium chloride in concentrated hydrochloric acid (100 cm^3 HCl (37 %) + 10 g NaCl electrolytic). Afterwards, the samples were exposed to electrolytic etching with an AC power supply (3–6 V). Because of the "forced corrosion", brought on by the etching, the specimen surface showed inhomogeneous corrosion, and different microstructural features, which varied from area to area, were revealed.

3 RESULTS

The results of the resistivity measurements are plotted in **Figure 4**. Because the plot was difficult to interpret, a transformation of the plot was performed, in which the first derivative of the resistance-change plot was used to identify the points where the resistance change was most apparent.

It can be seen in **Figure 4** that immediately upon heating from room temperature, the electrical resistance of the strips linearly increased with increasing temperature. The starting conditions were as follows: temperature 27 °C and resistivity 20.846 × 10^{-3} Ω. At approximately 280 °C, a small decrease in the resistivity was observed, which could have been the result of a decrease in the dislocation density as part of the recovery. Nevertheless, above 500 °C, a larger decrease in resistivity was recorded, and a maximum decrease was attained at approximately 750 °C. This is consistent with the recrystallization trend (dislocation-free grains), in which the corresponding maximum peak was determined by differential scanning calorimetry (not shown in this paper).

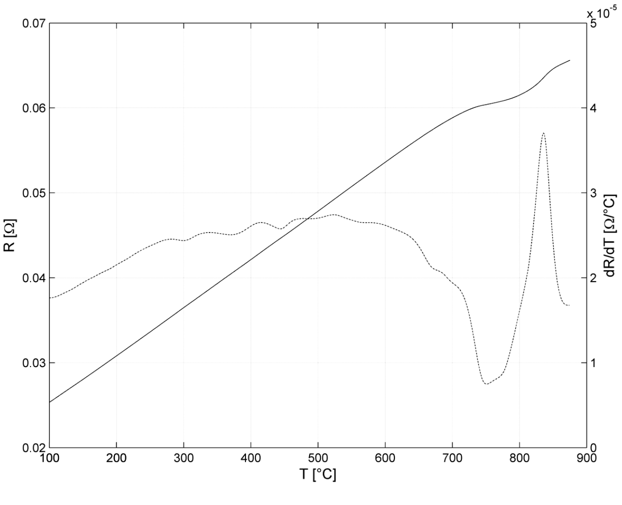

Figure 4: A plot of the resistivity changes during the recrystallization versus the temperature

Slika 4: Diagram spreminjanja upornosti med rekristalizacijo v odvisnosti od temperature

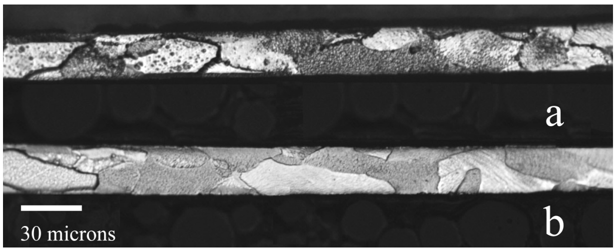

Figure 5: Microstructure: a) deformed and b) recrystallized

Slika 5: Mikrostruktura: a) deformirano, b) rekristalizirano

The recrystallization is shown to depend on the stored energy of the cold-worked foil, the nature of the nucleation sites, and the pinning of the boundaries. Furthermore, knowing that the hardness of platinum and platinum alloys decreases with the increasing temperature and time of annealing, a rapid decrease in the hardness is expected in the temperature range where the recrystallization takes place.[39] According to Loginov et al.,[24] with a decrease in the initial deformation, the annealing point shifts towards higher temperatures.

According to a comprehensive study about the work-hardening, recovery, and recrystallization of three grades of platinum, reported by Raub (1964),[40] pure platinum was recovered after heavy cold-working at approximately 200 °C. The temperature ranges for the recovery and recrystallization for platinum of different grades, however, were found to increase with increasing impurity content.

An optical micrograph of cold-rolled and recrystallized strips is shown in **Figures 5a** and **b**, respectively. The etched specimens showed a selectively corroded surface as a consequence of different grain orientations, crystal defects such as dislocations and grain boundaries, and cold-worked regions. The grains appear as elongated particles when the differential interference-contrast technique was used. A finer structure is observed in the cold-rolled sample (**Figure 5a**). After a short period of recrystallization, slightly larger grains are observed (**Figure 5b**). The size of the grains depends not only on the temperature used but also on the annealing time. In this study, the relatively small increase of grain size after annealing confirms the benefit of using a low annealing temperature and a short annealing time.

4 DISCUSSION

The present work aims at giving some explanations about the microstructure of platinum foil to be used in the fabrication of electrodes for SNS, in both the cold-rolled hardened and annealed conditions. More specifically, this paper discusses an analysis, the design criteria, and the structural properties of the platinum ribbon to be used for the fabrication of capacitive as well as faradaic recording and stimulating electrodes within the multi-electrode spiral nerve cuff for SNS.[9] Namely, it is important to understand all the problems that may occur when using an electrode for SNS and the situations in which a stimulating electrode poses the greatest risk to the nerve.[6] Only by combining the knowledge about appropriate stimulation parameters and the thermal treatments of cold-worked platinum foil, could result in the optimized fabrication of SNS electrodes used in clinical studies. Namely, it is well known from the literature that significant chemical reactions may occur during stimulation that can destroy a platinum stimulating electrode.[41] Decomposition of the stimulating electrode is a far more significant problem than just the loss of the electrode itself. It can result in the deposition of metal ions in the body of the patient.

5 CONCLUSION

To address some of the key issues currently inhibiting the widespread adoption of implanted stimulation technologies, research in the design and characterization of metallic materials for implantable multi-electrode systems should be intensified. To achieve suitable final properties of the platinum foil for the fabrication of stimulating electrodes, it is crucial to establish the right combination of plastic deformation and annealing treatment. From these experiments, we concluded that the deployed measurement technique is adequately sensitive to the resistivity changes due to the cold-rolling so that the temperature of recrystallization could be determined. Besides the aforementioned techniques used, however, a wide variety of analytical techniques such as Auger and SEM, could be used alongside it to provide a far more complete knowledge of the microstructure. In this regard, the direction of our further work would involve quantitative and qualitative information about the physical and chemical changes during the cold deformation of a platinum foil that would be obtained using the thermo-analytical technique of differential scanning calorimetry. Furthermore, the recrystallization kinetics of cold-rolled platinum foil containing an oriented grain structure will be studied.

In summary, this study utilized electrical resistivity measurements to characterize a material with the potential for use in the production of neural stimulation electrodes.

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