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Research Article

Forecasting of Corrosion Properties of Steel Wires for Production of Guide Wires for Cardiological Treatment

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The study presents evaluation of the influence of strain in drawing process and of surface modification on resistance to electrochemical corrosion of wires made of stainless steel for production of guide wires used in invasive cardiology. The results of static tensile test enabled us to determine the course of flow curve of wires made of X10CrNi 18-8 steel as well as mathematical form of flow stress function. Resistance to electrochemical corrosion was evaluated on the ground of registered anodic polarisation curves by means of potentiodynamic method. The tests were performed in solution simulating human blood on samples that were electrolytically polished and samples that were polished and then chemically passivated. Exemplary anodic polarisation curves were given. It was proved that with the applied strain, corrosion properties decrease. It was found that chemical passivation improves wire corrosion characteristics. Statistical analysis showed that there is a significant dependence between corrosion properties (polarisation resistance R_p) and strain ε applied in drawing process. Functions that present the change $R_p = f(\varepsilon)$ were selected. The issue is of importance to guide wire manufacturers because application of the suggested methodology will enable us to forecast corrosion characteristics of wire with the required strength drawn with the applied strain.

1. Introduction

Guide wires are indispensable instruments used in low-invasive treatment. Guide wires were initially manufactured for vascular applications, but over time they became indispensable also in other branches of medicine. They can be used in treatment where percutaneous access inside blood vessels is required—in angioplasty, in electrocardiology, when inserting electrodes to the heart, and in endoscopic gastrostomy or endourology [1–3]. In the latter one they are used for two purposes: (1) they ensure access to specific parts of urinary system and (2) they are used as guide wires over which catheters and stents can be inserted.

Such a wide application spectrum determined production of many different types of guide wires. They differ from one another in construction, dimensions, material they are made of, and mechanical characteristics, including stiffness or resistance to cracking [1]. Manufacturers make guide wires in their own catalogue series of types. Most of them feature elastic flexible tip and stiff or semistiff body. Elastic tip enables us to avoid tissue damage and perforation. Most frequently, guide wires have the form of long, thin straight wires or wires with "J"-shaped tip. Also guide wires with more complicated construction have found their application. For example, in endourological percutaneous nephrolithotripsy or ureterorenoscopy, guide wires consist of both round wires and springs in which there are thin round wires and flat wires [4–8].

In invasive cardiology, thanks to application of intravascular guide wires, it is possible to insert the required instruments into blood vessels or, for example, implants, stents used in angioplasty. During treatment there is no need to cut blood vessels. In electrotherapy, guide wires are used in two ways. The first one includes accessing the vein, thanks to which an electrode can be inserted. The second one includes stiffening of the electrode, which facilitates its insertion in the respective place inside the heart.

Guide wires must meet a number of requirements. Most of all, their application must be safe for the patient. They should feature high resistance to electrochemical corrosion in the environment of tissues and saline solutions. When coming into contact with blood, they cannot cause any reaction. Guide wires should also feature the required mechanical properties suitable for the respective application. Functional properties are dependent on, among other things, chemical composition of the material, its metallurgical purity, and technological parameters of wire production process. The question of determination of physical and chemical characteristics of guide wire surface is also vital. They should be adapted to the characteristics of human tissue environment.

In case of plastic working, the selection of optimum parameters of wire drawing process is influenced by proper characteristics of technological plasticity of the material. It conditions obtaining the structure susceptible to drawing process and obtaining a product that features the required functional characteristics. Plastic strain is accompanied by the phenomenon of work hardening that is connected with the increase of flow stress σ_p. Hardening causes increase of strength properties of wire and decrease of its plastic properties. Correct determination of parameters of plastic working and obtaining suitable final properties of products are connected with analysis of the course of function $\sigma_p = f(\varepsilon)$, where ε means strain expressed as a logarithm. Curves of flow stress change as the function of strain (so-called flow curves) enables us to forecast behaviour of the material during plastic working [9]. Strain applied in drawing process also has a significant influence on corrosion properties of wire used for production of guide wires [10, 11].

Proper solution to technological problems regarding production of guide wires conditions the success of performed treatment in invasive cardiology. Engineers make use of work-hardening curves in order to select such parameters of drawing so as to obtain wires with mechanical characteristics required for the respective application. This study is a proposal of a similar behaviour in order to forecast corrosion characteristics of wire depending on strain applied in drawing process. Methodology presented in this study will enable us to define corrosion properties featured by the wire with the required strength drawn with the selected strain.

Guide wires in commercial use are manufactured among other things from stainless steel of X10CrNi 18-8 grade. The study presents the course of flow curve of wires made of that steel and mathematical form of flow stress function. Resistance to corrosion was evaluated on the ground of registered anodic polarisation curves by means of potentiodynamic method. Tests were made in the solution simulating human blood on samples that were electrolytically polished and polished and then chemically passivated. Exemplary anodic polarisation curves were presented, as well as curves showing the relation between polarisation resistance as the function of strain applied in wire drawing process. Performed tests enabled us to determine both the effect of strain during cold working and the way of surface preparation on wire corrosion characteristics.

2. Materials and Methods

Initial material for tests consisted of annealed wire rod made of X10CrNi 18-8 steel with diameter of 5.65 mm. Wire rod was drawn to a diameter of 1.5 mm. Total logarithmic strain in drawing process was $\varepsilon = 2.65$. Wire work hardening took place during cold working. In the course of strain process samples for mechanical and corrosion tests were cut off.

Flow stress σ_p of drawn materials is determined on the ground of stress-strain curve in tension in the arrangement σ_r-ε, where σ_r is real stress and ε is strain expressed as a logarithm. In order to determine actual stress, it is necessary to calculate real cross-section of sample S loaded with force F. Calculation of real cross-section of sample requires determination of real diameter of sample d, which is subject to change due to present elastic and plastic elongation. For the load corresponding to proof stress, the value of real stress $\sigma_{r0.2}$ was calculated in accordance with the formula (1) [9]

$$\sigma_{r0.2} = \frac{R_{p0.2}}{\left[\sqrt{1/1.002} - \nu \cdot R_{p0.2}/E\right]^2}, \tag{1}$$

where $R_{p0.2}$ is proof stress, ν is Poisson's ratio, and E is Young's modulus.

Strength properties, including proof stress, were determined in static uniaxial tension test on testing machine Instron of 1116 type. Next, the course of flow curve of the function $\sigma_p = f(\varepsilon)$ was determined.

Wires were then subject to surface modification. Wires electropolished as well as polished and then chemically passivated in 40% nitric acid surface were selected for tests. Passivation proceeded at the temperature of 65°C for 20–60 min. Prior to polishing and passivation, wires were grounded with abrasive paper with granulation from 80–1200. It enabled us to remove subgrease layers and grease that was left on the surface after drawing process. Figure 1 shows pictures of wire rod and wire with diameter of 1.5 mm after drawing. On the surface, there is a visible subgrease layer (wire rod) and a layer of grease (wires). If greases had been left there, it would preclude correct execution of electrochemical polishing and passivation. It would also cause deterioration of corrosion characteristics of wires. Pictures of wire surface after plastic working were taken with electron scanning microscope with field emission FE SEM S-4200 Hitachi.

Resistance to electrochemical corrosion was evaluated on the ground of registered anodic polarisation curves by means of potentiodynamic method [12, 13]. Potentiodynamic tests of wires were performed in artificial blood plasma solution. Stern method was used in order to determine polarisation resistance. Potentiodynamic tests were performed at the temperature of $T = 37 \pm 1$°C and pH $= 7.0 \pm 0.2$. Saturated calomel electrode (SCE) of KP-113 type served as reference electrode, whereas platinum electrode PtP-201 was used as auxiliary electrode. Table 1 presents composition of artificial blood plasma.

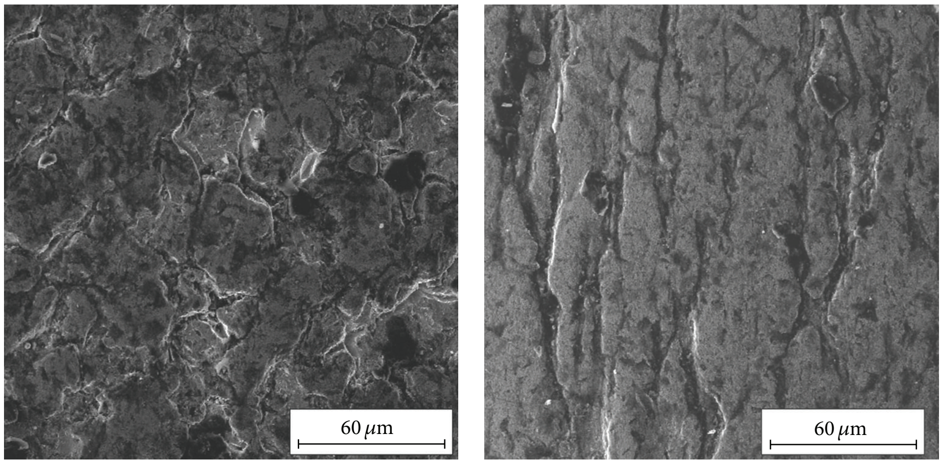

Figure 1: Surface of wire rod (a) and wire with diameter of 1.5 mm (b).

Table 1: Chemical composition of artificial blood plasma solution.

Chemical compound	Amount of distilled water, g/L
NaCl	6.8
$CaCl_2$	0.2
KCl	0.4
$MgSO_4$	0.1
$NaHCO_3$	2.2
Na_2HPO_4	0.126
NaH_2PO_4	0.026

3. Results

Table 2 presents strength properties of wires with selected diameters determined in tensile test.

Real stress values $\sigma_{r0.2}$ determined in tensile tests, corresponding to proof stress, served as the ground for making flow curve of tested wires and determination of mathematical form of flow stress function. The curve was approximated with function $\sigma_p = \sigma_{p0} + C\varepsilon^n$ that gives consideration to the value of stress for initial state (i.e., for annealed wire for drawing). Mathematical form of flow stress function for the tested steel is as follows:

$$\sigma_p = 253.1 + 894.6\varepsilon^{0.51}. \quad (2)$$

Figure 2 presents flow curve of wires made of X10CrNi 18-8 steel (ε means strain in drawing process expressed as a logarithm).

Potentiodynamic tests in artificial blood plasma enabled us to determine how wire resistance to electrochemical corrosion changes both in relation to strain applied in drawing process and to the way of wire surface preparation. OCP potential for all tested samples stabilised after 60 minutes. Corrosion test results are shown in Table 3. Figures 3 and 4 present selected anodic polarisation curves.

Next, it was determined whether there is a significant relation between corrosion properties and strain in drawing process. Figure 5 shows curves obtained on the ground of selected results of corrosion tests, namely, the change of polarisation resistance R_p as the function of strain applied in drawing process ε. Functions presenting the change $R_p = f(\varepsilon)$ were selected. They look as follows:

(i) electrochemically polished wires

$$R_p = -118.4\varepsilon + 592.5, \quad R^2 = 0.886, \quad (3)$$

(ii) wires that were polished and chemically passivated

$$R_p = -444\varepsilon + 2057.5, \quad R^2 = 0.842. \quad (4)$$

Static analysis proved that significance level $P < 0.05$.

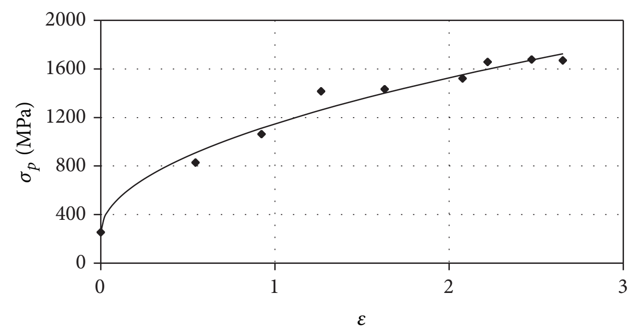

Figure 2: Flow curve of wire made of X10CrNi 18-8 steel.

4. Discussion

Experimental determination of flow stress of the material is executed in one of basic tests: tensile, compression, or torsion. Indeed, static tensile test is accompanied by strain too small to be taken into consideration by engineers who design plastic forming processes; still it is the only possible one to be adopted in case of tests of thin wires. Performed mechanical

Table 2: Strength properties of wire.

Wire diameter d, mm	Logarithmic strain in the drawing process, ε	Tensile strength R_m, MPa	Proof stress $R_{p0.2}$, MPa
5.65	—	604	252
3.0	1.27	1607	1403
2.0	2.22	1827	1507
1.5	2.65	2178	1653

Table 3: Test results of pitting corrosion resistance.

Wire diameter d, mm	Strain in drawing process ε	Breakdown potential E_b, mV	Polarisation resistance R_p, kΩcm^2	Corrosion current density i_{corr}, μA/cm^2
Electrochemically polished wires				
5.6	0	+612	567	0.04
3.0	1.27	+510	459	0.10
1.5	2.65	+380	229	0.11
Electrochemically polished and chemically passivated wires				
5.6	0	+858	2220	0.012
3.0	1.27	+778	1230	0.021
1.5	2.65	+695	1070	0.024

properties tests enabled us to determine the course of flow curves and to select flow stress function of drawn wires made of X10CrNi 18-8 steel.

Performance of potentiodynamic tests enabled us to evaluate the impact of strain in drawing process on wire resistance to electrochemical corrosion artificial blood plasma solution. It was proved that with the applied strain, corrosion properties decrease. Perforation potential and polarisation resistance decrease, and anodic current density increases. That tendency applies both to electrolytically polished and polished and then chemically passivated wires.

Annealed wire rod with diameter of 5.65 mm featured the highest corrosion resistance, irrespective of the condition of the surface. Perforation potential of polished wire rod was E_b = +612 mV and, that of polished and passivated was E_b = +858 mV. The lowest perforation potential was observed in wire with diameter of 1.5 mm (E_b = +380 mV for polished wire and E_b = +695 mV for passivated wire). As strain increased, polarisation resistance also decreased. Polarisation resistance for polished wires was, respectively, R_p = 576 kΩcm^2 (wire rod) and R_p = 229 kΩcm^2 (wire with diameter of 1.5 mm). Polarisation resistance of polished and passivated wire rod was R_p = 2220 kΩcm^2 and of polished and passivated wire with diameter 1.5 mm was R_p = 1070 kΩcm^2.

Plastic strain in drawing process caused increase of corrosion current density for both types of wires, polished and polished and then passivated. Corrosion current density of polished wire rod was i_{corr} = 0.04 μA/cm^2 and of polished and passivated was i_{corr} = 0.012 μA/cm^2. Wire with diameter d = 1.5 mm featured the highest corrosion current density. It was i_{corr} = 0.11 μA/cm^2 for polished wire and i_{corr} = 0.024 μA/cm^2 for passivated wire.

Presented results show explicitly that chemical passivation substantially improved wire resistance to electrochemical corrosion in artificial blood plasma. Corrosion properties of passivated wires are higher than those of wires that were subject only to electrolytic polishing.

Static analysis showed that there is a significant dependence between corrosion properties (polarisation resistance) and strain applied in drawing process. Presented curves and functional relations give the ground to conclusions related to polarisation resistance of wire subject to surface treatment. The value of polarisation resistance shows that it was justifiable to use chemical passivation in case of wires for production of cardiologic guide made of X10CrNi 18-8 steel.

Presented curves represent correctly the results obtained experimentally. The greatest differences of polarisation resistance can be observed for wire with initial diameter. It must be highlighted that functions presented in the paper were elaborated for one heat. To transform them into universal relations for stainless steel X10CrNi 18-8, it is necessary to repeat the tests for a bigger number of heats.

5. Summary and Conclusions

The study proves that increase of strain in drawing process of wires made of stainless steel X10CrNi 18-8 causes decrease of their resistance to electrochemical corrosion in artificial blood plasma. Moreover, it was observed that chemical passivation improves wire corrosion characteristics.

Potentiodynamic test results also enabled us to obtain functional relations showing the influence of strain in drawing process on the change of polarisation resistance. It must

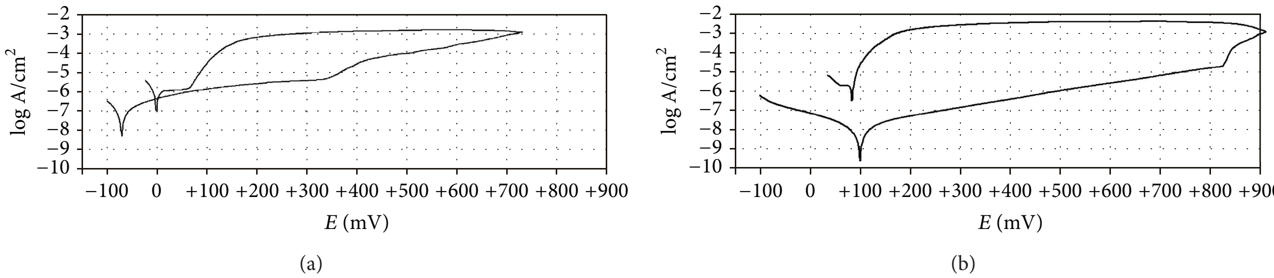

Figure 3: Anodic polarisation curve recorded for wire rod (d = 5.65 mm) electropolished (a) and passivated (b).

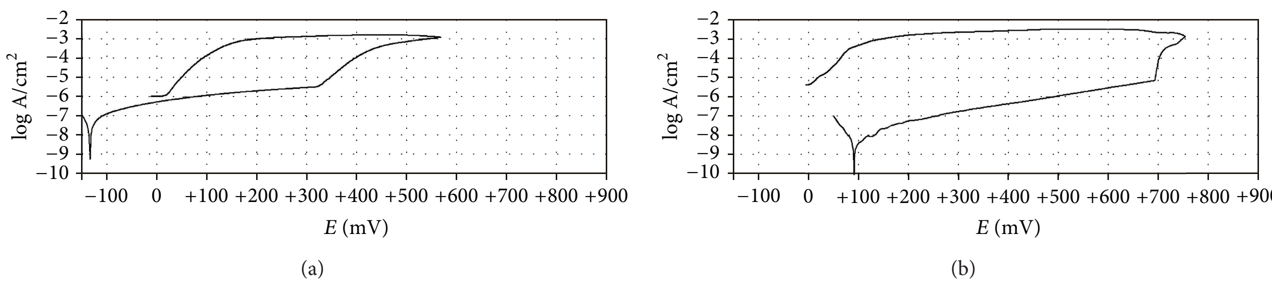

Figure 4: Anodic polarisation curve recorded for wire d = 1.5 mm electropolished (a) and passivated (b).

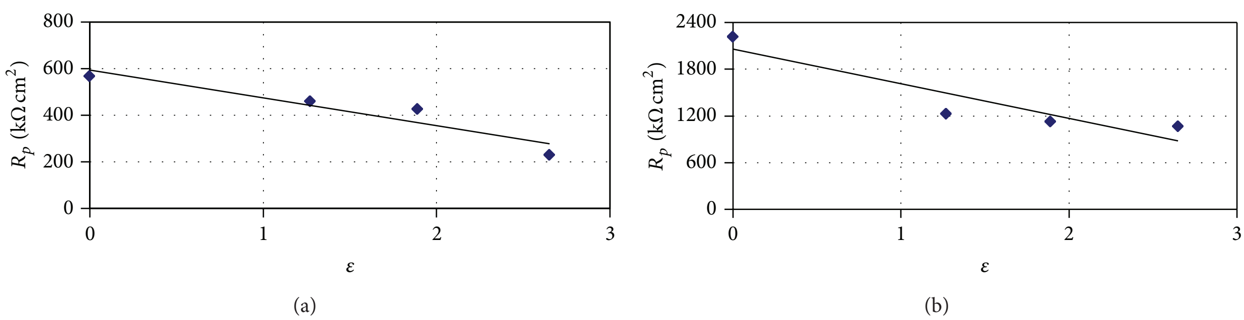

Figure 5: Dependence of polarisation resistance on strain in the drawing process: (a) of electrochemically polished wire and (b) of electrochemically polished and passivated wire.

be mentioned that presented functional relations are not of universal character and refer only to wires made of X10CrNi 18-8 steel tested in artificial blood plasma. If they are to be applied either to wires or other materials (e.g., other grades of steel, titanium alloys, cobalt alloys, alloys with elastic memory effect, and many other) or goods for other purposes, it is necessary to perform similar test for wires made of the respective material in another environment simulating human body saline (artificial urine solution, artificial saliva, and Tyrode or Ringer solution).

Problematic aspects presented in the study are crucial for process engineers who deal with designing wire drawing processes for medicine, as the issue of correct description of material plasticity is tightly connected with the selection of optimum parameters of wire production technological process. That issue is equally important to guide wire manufacturers, because if the respective curves or functions are to be applied, it is possible to forecast in advance corrosion characteristics of wire with the required strength drawn with the applied strain.

To sum up, it must be highlighted that proper description of flow stress function is one of the elements that determine acquisition of proper characteristics of technological plasticity of materials. These characteristics constitute currently the ground for databases for computer simulation of plastic working processes. Such bases could be completed with functional relations $R_p = f(\varepsilon)$ determined for various metallic biomaterials adjusted to human tissue environment in various body saline solutions. Then they could be used by process engineers and manufacturers of goods produced by means of cold working for various medical purposes. For example, when manufacturing a product (strip, bar, and wire, etc.) with the required mechanical characteristics, database will enable us to read both the value of necessary strain applied in processing and polarisation resistance corresponding to such strain that the respective biomaterial will feature.

That innovative proposal regarding creation of databases which take into account corrosive properties of metallic materials for medicine would make an extremely useful tool in designing technological processes of biomaterials.

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