EVALUATION OF THE DIFFERENTIATION OF STRUCTURAL AND PHYSICO-CHEMICAL PROPERTIES OF ORTHODONTIC WIRES OF AISI 304 STAINLESS STEEL

Dominika Grygier[1], Victoria Hoppe[2], Anna Zięty[3], Małgorzata Rutkowska-Gorczyca[1]

[1] Wrocław University of Science and Technology, Department of Materials Science, Strength and Welding Technology, Poland
[2] Wrocław University of Science and Technology, Scientific Association of Material Science of the Nation doc. Rudolf Haimann, Poland
[3] Wrocław University of Science and Technology, Department of Mechanics, Materials Science and Engineering, Poland
*e-mail: dominika.grygier@pwr.wroc.pl

Abstract

Wires used for orthodontic arches play a very important role in the process of orthodontic treatment. In combination with the lock attached to the tooth, they move and align the teeth along the set trajectories. Wires of stainless steel are commonly used in orthodontics for several reasons: they are characterised by high resistance to corrosion, high strength and elasticity, formability and a possibility of obtaining defined properties through cold working and annealing during production process. The purpose of the research presented in the work is the analysis of differences of the selected structural properties in the context of corrosion resistance of the orthodontic wire material. The object of the research was edge arches of the 0.016"x0.022" size made of the stainless steel type AISI 304, provided by two different producers: G&H Orthodontics and Adenta. The research methodology involved analysis of chemical and phase composition of the tested alloy, microscopic tests with application of the light and electron microscopy methods, as well as electrochemical direct current measurements.

The research presented in the work has shown significant differences in structural and physical-chemical properties of the orthodontic wires made of AISI 304 type stainless steel. Despite the fact, that the tested arches were manufactured of the theoretically the same materials, but by different producers, they significantly differ in chemical composition, metallurgical purity, phase composition and corrosion resistance. In addition, it is worth noticing that the tested materials, in terms of structure, do not meet the normative requirements obligatory for biomaterials.

Keywords: *stainless steel, orthodontics wire, microstructure*

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Introduction

Orthodontics is one of the fields of dentistry, dealing with the diagnosis, prevention and correction of malocclusion [1-3]. Orthodontic treatment is primarily aimed at correcting the appearance of dentition, facial features, but also restoring those activities that were incorrectly performed due to incorrect dentition, e.g. chewing food or correct pronunciation. Orthodontic tooth displacement results mainly from the application of force to the teeth [2]. The forces generated by orthodontic appliances are selected and activated by the orthodontist. Teeth, as well as supporting structures, react to exerted force through various biological responses and lead to tooth displacement in the supporting bone. Proper application of the principles of biomechanics leads to an increase in the efficiency of treatment through the improvement of planning and carrying out orthodontic care.

One of the popular methods of treating malocclusion is the orthodontic fixed braces. The main elements of the apparatus are wires, called arches and fastening elements [3,4]. Fixing elements include rings, ligatures and various types of locks, fixed directly to the tooth surface. However, the most important element of the apparatus is the arch. The role of the orthodontic arch in the treatment process is both as a spring and as a conductor. The force that is required in the process of deflecting the wire to the slit of the lock, causes a certain amount of work, called the activation energy that causes the tooth to move [3,5]. Wires used for orthodontic arches have many applications in the orthodontic treatment process. In conjunction with the lock attached to the tooth, they are to move and align the dentition along the set trajectories. Wires with larger cross-sections serve as retainers, which are supposed to prevent the return of teeth to the original arrangement in the arch [4]. Materials commonly used for orthodontic wires are austenitic stainless steel of the AISI 302 and AISI 304 grades, nickel-titanium alloys called Nitinol, beta-titanium and cobalt chrome alloys [5-7]. Each of these types of orthodontic wires, due to its specific, desirable properties, is used in various stages of orthodontic treatment. Stainless steel wires are widely used in orthodontics for several reasons: they are characterized by high corrosion resistance, high strength and elasticity, formability and the ability to achieve specific properties by cold working and annealing during the production process, as well as low manufacturing costs. These materials also meet the condition of biocompatibility in tissues and fluids of the stomatognathic system, have the required high corrosion resistance, permanent aesthetic features and specific organoleptic properties [5,8-10]. AISI 302 stainless steel is austenitic steel, containing from 17% to 19% chromium, 8% to 10% nickel and about 0.15% carbon, is non-magnetic, extremely durable and plastic. It is one of the most popular chromium-nickel and stainless steels. Cold working dramatically increases its hardness, and the range of applications ranges from stamping, broaching and forming wire to moulding in various types of washers, springs and plates. AISI 304 stainless steel is a non-magnetic alloy containing from 18% to 20% chromium, from 8% to 12% nickel and a maximum of 0.08% carbon content. The steel of this grade has less carbon, which minimizes the precipitation of carbide precipitates. It is widely used in the production of equipment in the mining, chemical, cryogenic, food, dairy and pharmaceutical industries. High corrosion resistance in acidic environments makes 304 stainless steel ideal for medical applications. Stainless steel orthodontic wires began to be used already in the 1920s. The wire manufacturing processes improved its properties and enabled the production of various wire shapes, which in time convinced sceptical orthodontists.

A few years later Begg started using stainless steel round wires, and in the early 1940s, he started working with Wilcox, to produce a different type of stainless steel wire: the Australian stainless steel. Stainless steel has not been completely accepted in the physicians' environment, to the point where Archie Brusse (founder of Rocky Mountain Metal Products) presented a few decades later in 1933 at the American Society of Orthodontics, complete clinical treatment on the first system made of stainless steel, which resulted in the fact that by 1950, the 300 series was used in most orthodontic materials [11-13].

Materials and Methods

Purpose of the research presented in the work is an analysis of differentiation of the selected structural properties in the context of corrosion resistance of the material of the orthodontic wire. The object of the research was edge arches of the 0.016”x0.022” size made of the stainless steel type AISI 304, provided by two different producers: G&H Orthodontics and Adenta.

The X-ray microanalysis of the tested orthodontic wires was performed with the use of the SEM-EDS method. The quantitative and qualitative analysis was performed with the use of Zeiss EVO Ma25 scanning electron microscope equipped with the EDS Quantax probe from Bruker. The measurements were conducted with the accelerating voltage of 20 kV at the areas of 110 μm x 150 μm, at the counting time 500 s. The percentage of carbon and sulphur in the steels was determined additionally using the Leco CS-444 carbon and sulphur analyser, which uses the HF-400 induction furnace and measures carbon and sulphur by infrared absorption. Phase analysis studies were performed using an Ultima IV Rigaku X-ray diffractometer, in the range of angles 2θ from 40 to 90 with a step of 0.05. The resulting diffractograms were analysed using FindIt software using the Inorganic Crystal Structure Database Fiz Karlsruhe. The obtained results were presented using the OriginLab software. The assessment of the degree of contamination of steel with non-metallic inclusions was made in accordance with the ISO 4967: 2013 standard, comparing the recorded metallographic specimens of wires in the non-etched condition, with a magnification of 100x with the standards included in the standard. The microstructure observations were conducted using the NIKON ECLIPSE MA200 light microscope with the use of NIS Elements BR software, image recording was performed using the CCD Nikon DS.-Fi1 camera at magnifications: 100x, 200x and 500x. The microscopic tests were performed in the etched and non-etched states. As the etching agent, the 10% oxalic acid was used. The samples were electrolytically etched for 90 s with the 5.4 V voltage and 1.4 A current.

Determining the average number of carbide grains in the microstructure was performed with the application of quantitative metallography and the Jeffries's method. The Jeffries's method enables determining the average number of grains or precipitations at 1 mm^2 of the surface. A circle with a diameter of 79.8 mm and a surface of A = 0.5 mm^2 should be applied to the photo taken of the microstructure. Next, the number of grains or precipitations lying completely inside the NW circle and the number of grains or precipitates cut by the N_i circle is calculated, where after the total number of grains or NT precipitations on the surface of circle A is calculated:

$$N_T = N_w + kN_i \quad (1)$$

where:
k – usually assumed as equal to 0.5.

The corrosion tests were performed with the use of the three-electrode system – the working electrode (WE), created by a fragment of wire applied for orthodontic wire of the AISI 304 steel, calomel reference electrode (RE) and the platinum counter electrode (CE). For the measurements, the ATLAS 1131 potentiostat from EU&IA was applied. The corrosion resistance of the materials applied for orthodontic wires was evaluated at the base of the polarisation curves run performed with the potentiodynamic method in the Ringer solution – $NaCl + KCl + CaCl_2 \cdot 6H_2O$ – (8.6 mg + 0.3 mg + 0.33 mg)/ml, at the room temperature. The polarisation curves were recorded for the rate of potential change equal to 1 mV/s.

Results and Discussions

The tests of the chemical composition of the orthodontic wires were performed with the use of the SEM-EDS technique (TABLE 1). The content of phosphorus, sulphur, chromium and nickel tested in the orthodontic arches of the AISI 304 steel does not exceed the values specified in the standard. The content of chromium and nickel oscillates within the limits of minimum values given by the standard. Due to the fact, that nitrogen and carbon are light elements and are not easily detected in the EDS method, it was decided to determine the carbon content by another method. The studies have shown that for light elements such as sulphur and carbon much better method of quantitative analysis of the elements is the method of combustion with the use of the infrared detector. The content of carbon in the tested arches slightly exceeds the permissible content given by the standard, however, this is the value in the 3rd decimal place, which can be considered as an admissible measurement error (TABLE 2). Orthodontic wires have an insignificant amount of sulphur in its chemical composition.

TABLE 1. Comparison of alloy element contents in the tested orthodontic wires with reference to the standard AISI 304 [14].

	AISI 304		G&H Orthodontics	Adenta
	wt.%	max	wt.%	wt.%
Si	1.00		0.83	1.10
Mn	2.00		2.10	2.16
P	0.045		0.000	0.000
S	0.030		0.030	0.010
N	0.11		0.00	0.01
Cr	17.0	19.5	17.85	17.86
Ni	8.00	10.50	8.64	8.56

TABLE 2. Percentage contents of carbon and sulphur in the tested samples in reference to the AISI 304 standard [14].

AISI 304		G&H Orthodontics		Adenta	
chemical element	max wt.%	[g]	wt.%	[g]	wt.%
C	0.07	0.2752	0.07631	0.2946	0.07641
S	0.03		0.00061		0.00195

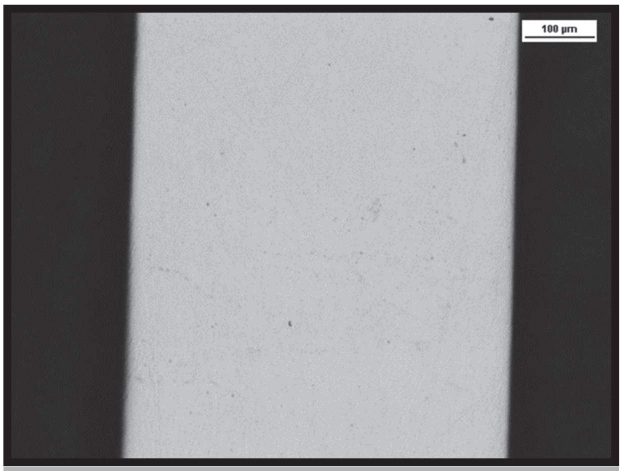

FIG. 1. Metallographic section of G&H Orthodontics orthodontic wire. Non-Etched state. LM

FIG. 2. Metallographic section of Adenta orthodontic wire. Non-Etched state. LM

FIG. 3. The microstructure of the G&H Orthodontics orthodontic wire. Visible fibrous texture and precipitations of the $Cr_{23}C_6$ carbide arranged in bands. Etched state. LM

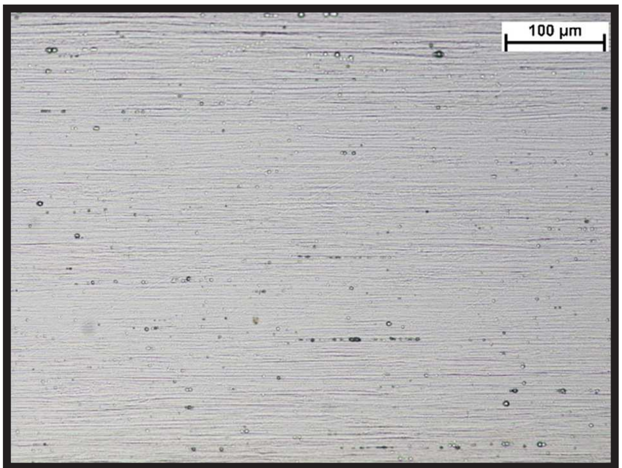

FIG. 4. The microstructure of the Adenta orthodontic wire. Visible strongly crushed structure with precipitations of the $Cr_{23}C_6$ carbide. Etched state. LM

Microscopic studies of the tested orthodontic arches material in the non-etched state have shown the appearance of non-metallic inclusions in the form of oxides in the quantity not exceeding the standard number 2 according to the PN-64/H-04510 standard (FIGs. 1 and 2). Such large number of the non-metallic inclusions is not allowed in materials for medical applications. Moreover, the type of inclusions, their shape, quantity and the way of their deployment can have a huge impact on the anisotropy of the mechanical properties of the material [8,10]. Mechanical properties of the orthodontic wires are of great importance because they are the main factor determining the effectiveness of treatment [2,10].

Microscopic studies in the etched state have shown the presence of strongly deformed austenite structure, being the result of the arches forming process by cold drawing (FIGs. 3 and 4). The size and deployment of grains in the microstructure cannot be determined. In the microstructure of the tested wires, which is confirmed by subsequent studies, also the chromium carbide of the $Cr_{23}C_6$ type is found and noticeable in the form of spheroidal, arranged in bands precipitations between grains of the deformed austenite.

In order to determine the quantitative content of chromium carbide present in the tested material, the quantitative metallography method with the use of Jeffries method was applied (TABLE 3). The smallest number of the $Cr_{23}C_6$ type carbide is in the wire used for the Adenta orthodontic wire, amounting to some 1330 grains/1 mm^2. The G&H Orthodontics orthodontic wire has by 200 precipitations of the carbide more at the same surface.

TABLE 3. Quantitative metallography of the $Cr_{23}C_6$ type chromium carbide content with the use of Jeffries method.

Sample	N_i	N_w	N_T	k	N_A
G&H Orthodontics	5	47	49	0.5	1560
Adenta	4	40	42	0.5	1332

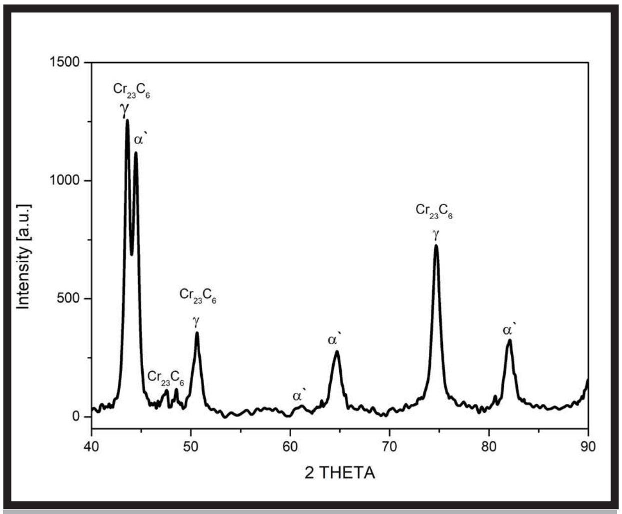

FIG. 5. X-ray radiation spectrum with marked reflexes coming from individual phases contained in the tested alloy of the G&H Orthodontics arch.

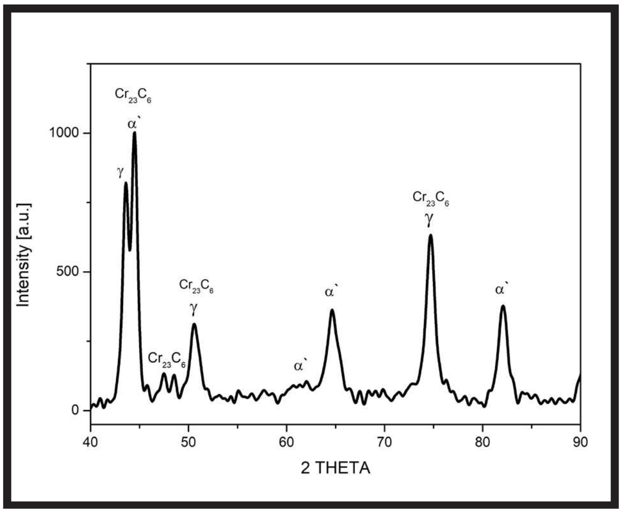

FIG. 6. X-ray radiation spectrum with marked reflexes coming from individual phases contained in the tested alloy of the Adenta arch.

The phase composition analysis studies using the XRD method have shown that materials of the orthodontic arches have a three-phase structure. The reflexes corresponding to the phase γ, α` and $Cr_{23}C_6$ have been identified (FIGs. 5 and 6). The primary phase of the type AISI 304 alloys is austenite (γ). As a result of the cold plastic working from the γ phase, the martensite induced by crushing is created, the so-called strain-induced martensite (α`). The $Cr_{23}C_6$ carbide is created as a result of annealing of the austenitic steel after cold plastic working at temperatures above 400°C.

During plastic straining of metals and alloys with the A1 lattice (regular wall centred) a microstructure is created, dependent mainly on such parameters as chemical composition, strain temperature and thermodynamic phase stability. The phase change taking place during plastic straining with the course γ→ε→α' or γ→α' causes strong material hardening, which can be verified by previously performed hardness tests. The bibliography gives that the AISI 304 steel in the plastically undeformed state has the structure of equiaxed grains of the γ phase, characteristic, and instead, after deformation of the steel with the crush of about 50% – the structure of elongated austenite grains with the α' martensite phase. The formation of the α' phase causes the fragmentation of the structure and, as a consequence, its strengthening [15,16]. While analysing the X-ray diffractograms, the intensity of reflexes coming from martensite and comparing the images of microstructure in the etched states it is assumed that the material crush significantly exceeded 80%, and the dominating phase in the studied materials is martensite - α'.

The higher values of the reflex coming from martensite indicate for the higher degree of the material crush [15]. The higher contents of the ferromagnetic phase α' will adversely affect the system, where orthodontic wires will stay possibly causing magnetothropism of blood components and form the corrosion centres. It is believed, that the carbide of the $Cr_{23}C_6$ type was created as a result of the metallurgy process and was not dissolved in the matrix, so that its small amounts remained in the orthodontic wire material, and the temperature of the annealing process after cold working exceeded 500°C, which causes precipitation of chromium carbide along the grain boundaries with simultaneous depletion of chromium within the grain [17].

Corrosion of orthodontic apparatus in the oral environment has for some time been a common topic among clinicians. These concerns focus on two main issues: whether the corrosion products, if they are manufactured, are absorbed by the body and cause local or systemic effects, and what are the effects of corrosion on the physical and clinical operation of orthodontic appliances. Due to the fact that the three-phase structure observed in the tested arches is particularly unfavourable because of the corrosion resistance, and the presence of the $Cr_{23}C_6$ carbide favours the intercrystalline corrosion, the electrochemical constant current measurements were performed and the potentiodynamic polarisation curves were determined.

At the base of the obtained study results it has been noticed that the orthodontic wires of stainless steel in the environment of the Ringer solution are not characterised by ability to passivation over the whole sample surface – no flattened current characteristics was observed at the graphs in the anodic range, which is confirmed by low abilities of the AISI 304 material in the above conditions to creating the stable passive layer (FIGs. 7 and 8). It has been observed that during exposal of the G&H Orthodontics orthodontic wire to the activity of the Ringer solution, the created passive layer is unstable, which increases the risk of ions flows favouring the development of the pitting corrosion. In the case of the Adenta orthodontic wire, the course of the curve indicates for typical pitting corrosion appearing at the material surface. The material of the arch No. 2 is characterised with the higher value of the corrosion potential (E_{corr} = -0.075 V) than the arch No. 1 (E_{corr} = -0.126 V), which indicates for better corrosion resistance (TABLE 4).

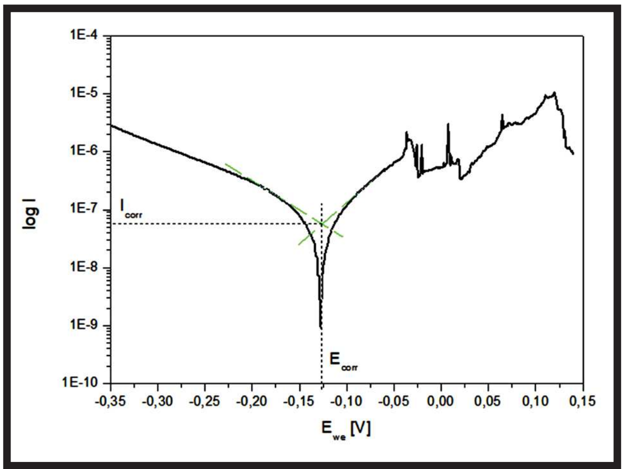

FIG. 7. The potentiodynamic polarization curve of the wire applied to the G&H Orthodontics orthodontic wire - the Ringer solution.

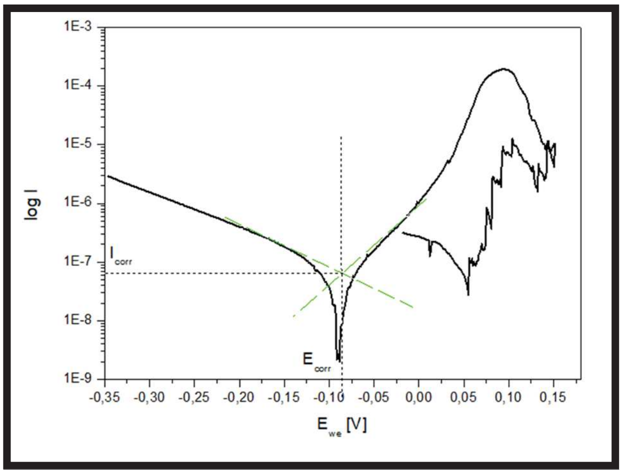

FIG. 8. The potentiodynamic polarization curve of the wire applied to the Adenta orthodontic wire - the Ringer solution.

TABLE 4. Summary of results of the polarization curve analysis (the Ringer solution).

Sample	E_0 (V)	I_{corr} (A/cm^2)	E_{corr} (V)
G&H Orthodontics	-0.117	$5.54\cdot10^{-8}$	-0.126
Adenta	-0.099	$7.05\cdot10^{-8}$	-0.075

Conclusions

The chemical composition tests have shown that the orthodontic wires from various producers differ in contents of individual alloy elements, as well as they exceed the contents of permissible manganese and sulphur values allowed by the standard. However, it could be stated, that the information provided by the producers that the wires are made of stainless steel of the AISI 304 type are confirmed.

Evaluation of the metallurgical purity degree has shown considerable diversity in terms of deployment and appearance frequency of non-metallic inclusions in the orthodontic wires from different producers. The research has shown that presence of the non-metallic inclusions in the tested materials is equal to the standard No. 2, according to the ISO standard, which is unacceptable for materials applied in the living body.

Microstructure of the tested wires applied for orthodontic arches have shown appearance of strongly deformed structure of austenite (the fibrous texture along the drawing direction). Between strongly deformed grains of austenite the clear initial etchings around other microstructures have been observed. The XRD analyses have shown occurrence of austenite in the microstructure of martensite induced by α' draft, as well as chromium carbide of the M23C6 type. The three-phase structure is particularly unfavourable due to the corrosion resistance, as presence of the $M_{23}C_6$ carbide in the tested material will favour the intercrystalline corrosion. Instead, presence of martensite, the ferromagnetic phase will adversely affect the body the orthodontic wires will be in, possibly causing magnetotropism of blood components and additionally be a source of corrosion.

The corrosion of orthodontic wires is closely related to the acidic environment of the mouth and the presence of fluoride ions, prophylactic agents and mouthwash solutions. Due to the thermic, microbiological and enzymatic properties aspects, the mouth environment is favourable in terms of biodegradation of metal and their alloys aspect, resulting in releasing metallic ions in the mouth. Along with the release of ions from metals or alloys the corrosion of orthodontic wires may lead to increase in surface roughness and their weakening, which can seriously impact the material strength, leading to mechanical damage or even fracture of the orthodontic materials. Based on the obtained test results of the orthodontic wires of the same geometry coming from two different producers it has been observed that in the environment of the Ringer solution they do not show the ability to passivate. At the polarization curves in the anodic area only the clear dissolution area is noticable. At the surface of the orthodontic wires also the phenomena of pitting corrosion take place, which is confirmed by the course the anodic curves. Moreover, it has been found that content of non-metallic inclusions and carbides occurring in the material microstructure definitely lowers corrosion resistance of the material.

The research presented in the work have shown significant differences in structural and physical-chemical properties of the orthodontic wires of the AISI 304 type stainless steel. Despite the fact, that the tested arches were manufactured of the theoretically the same materials, but by different producers, they significantly differ with chemical composition, metallurgical purity, phase composition and corrosion resistance. In addition, it is worth noticing that the tested materials, in terms of structure, do not meet the normative requirements obligatory for biomaterials.

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