The structural characterization of some biomaterials, type AISI 310, used in medicine

M G Minciuna1,2, P Vizureanu1,2*, C Hanganu3, D C Achitei1,2, D C Popescu4,  
S C Focsaneanu1  
1 Gheorghe Asachi Technical University, Faculty of Materials Science and Engineering, 61 D. Mangeron Blvd, 700050, Romania  
2 Center of Excellence Geopolymer & Green Technology (CEGeoGTech), School of Materials Engineering, Universiti Malaysia Perlis, 01000 Kangar, Perlis, Malaysia  
3 “Gr. T. Popa” Medicine and Pharmacy University, Department of Dental Medicine, 16 Universitatii Street 700115, România  
4 “Gr. T. Popa” Medicine and Pharmacy University, Department of Orthopedics and traumatology, 16 Universitatii Street 700115, România  
Email: mirabela.minciuna@yahoo.ro, peviz2002@yahoo.com, carmenhanganu1957@yahoo.com, dragos_adc@tuiasi.ro, dragospopescu1975@yahoo.com, focsaneanu.sergiu@gmail.com

Abstract. Orthopedics biomaterials are intended for implantation in the human body and substituted or help to repair of bones, cartilage or organ transplant, and tendons. At the end of the 20th century, the availability of materials for the manufacture implants used in medicine has been the same as for other industrial applications. The most used metals for manufacturing the orthopedics implants are: stainless steels, cobalt-chrome-molybdenum alloys, titanium and his alloys. The structural researches which are made in this paper, offer a complete analysis of AISI310 stainless steels, using: optical spectrometry, X-ray diffraction and scanning electronic microscopy.

1. Introduction
The orthopedics, dentistry, plastic surgery, ophthalmology, maxillofacial surgery, cardiology, neurology and neurology, and practically all the medical specialities require the biomaterials utilisation, in different goals like: diagnosis, prevention and therapy [1-4].

Metals are one of the most used biomaterials in the case of orthopaedic implants (figure 1), and not only, being known for his high usage resistance, high ductility and hardness. Most commonly used metals, for implants manufacturing, are the stainless steels, the cobalt-chrome-molybdenum alloys, the titanium and titanium alloys [5].

First stainless steel used like material for implant manufacturing was AISI302 steel, which have a resistance bigger than vanadium and is much resistant to corrosion. The steel based on vanadium is not used in implants because, actually, his corrosion resistance is inadequate [6].

*peviz2002@yahoo.com
Further, it starts to be use the Mo 18-8 stainless steel, which contain molybdenum and present an improved corrosion resistance in salted water. This alloy is known like AISI 316 stainless steel. In 1950, the carbon content from AISI 316 steel was reduced from 0.08% to 0.03%, fact that improves the corrosion resistance in chlorides. This new alloy is named AISI 316L [7].

![Orthopaedics implants](image)

Figure 1. Orthopaedics implants [8].

The metallic biomaterials are classified, in terms of chemical composition and physical structure, in 3 principal classes [9,11]:

- Technically pure metals
- Metallic alloys
- Composites with metallic matrix (CMM)

**Metals** are used in medical domain, curently, like surgical implants, dentistry materials and for manufacturing of various aparatus and medical devices. Exist must 30 metallic elements used in medicine like [12-13]:

- Implants and orthopedics and dentistry prosthesses: Fe, Al, Ti, Co, Cr, Mn, Mo, Nb, Ni, Sn, Ta, V, W, Zr.
- Nonprecious dentistry alloys, aloyed with: Co, Mo,Al, B, Be, Cd, Cr, Fe, Mn, Ni, Si, Ti, V, W.
- Precious and semiprecious dentistry alloy, aloyed with: Au, Ag, Cu, Ga, In, Ir, Pd, Pt, Rh, Ru, Sn, Ti, Zn.

**Alloys** used like surgery implants in orthopedics and dentistry, are clasified in:

- Austenite stainless steels – represents the alloys class base on iron, with a high content by chrome and nickel;
- Cobalt base alloys, named CoCr alloys, characterized by a high chrome content (25-30%), molibdenum (5-7%) and other metals like nickel, manganese, zirconium, tin;
- Titanic alloys, with 70-90% Ti, can contain elements like: Al, V, Nb, Ta, Mn, Zr and Sn.

The stainless steel is a iron-carbon alloy, aloyed with chrome, which have a high corrosion resistance in air, in salt and acids solutions. The stainless steels proccesed by plastic deformation are more types; these are clasified in 4 classes, in term of structure (according AISI – American Institute of Steel and Iron), like is present in table 1.

Class I – martensite stainless steels, which contain chrome like principal alloying element; these can be hardened by heat treatments with rapid cooling and formation of martensite [14]. The stainless steels from this class dont have enough corrosion resistance, but presents good mechanical properties. From this reason, are used in manufacturing of surgery instruments, mostly due the hardness properties obtained by martensite quenching [15].
Class II – ferrite stainless steels which have a structure formed by a Fe₉ solid solution and do not be hardened by heat treatment [16]. The steels from this class present good properties of corrosion resistance, especially to corrosion under load, being used in chemical and medical industry, mostly to manufacturing of recipients [17].

### Table 1. Plastically deformable stainless steels and their chemical compositions [18]

| Mark AISI | Chemical composition % | Mentions |
|-----------|-------------------------|----------|
|           | C | Cr | Ni | Other elements |        |
| **Class I – martensite steels** | | | | | |
| 410 | max 0.15 | 11.5 – 13.5 | - | - | Turbines, valvs |
| 420 | 0.35 – 0.45 | 12 – 14 | - | - | Surgery instruments |
| 431 | max 0.20 | 15 – 17 | 1.25 – 2.5 | - | High ductility |
| 440 | 0.60 – 0.70 | 16 – 18 | - | - | Extremely hard |
| **Class II – ferrite steel unhardened** | | | | | |
| 430 | max 0.12 | 14 – 18 | max 0.5 | - | - |
| 446 | max 0.20 | 23 – 27 | max 0.5 | max 0.25 N | Temperature resistant |
| **Class III – Cr-Ni austenite steels** | | | | | |
| 301 | max 0.15 | 16 – 18 | 6 – 8 | max 2% Mn | Hardened under load |
| 304 | max 0.08 | 18 – 20 | 8 – 12 | max 1% Si | Especial 18-8 steel |
| 304L | max 0.03 | 18 – 20 | 8 – 12 | max 1% Si | Low carbon content |
| 310 | max 0.25 | 24 – 26 | 19 – 22 | max 1.5% Si | Steel 25-20 hot resistant |
| 310X | max 0.08 | 24 – 26 | 19 – 22 | max 1.5% Si | Low carbon content |
| 314 | max 0.25 | 23 – 26 | 10 – 14 | 1.5 – 3% Si | Steel with Si and hot resistant |
| 316 | max 0.10 | 16 – 18 | 10 – 14 | 2 – 3% Mo | Steel 18-8 with Mo used for implants |
| 316L | max 0.03 | 16 – 18 | 11 – 14 | 2 – 3% Mo | Steel with low carbon content, used for surgery implants |
| 317 | max 0.08 | 18 – 20 | 11 – 14 | 3 – 4% Mo | Steel with a high molibdenum content |
| 321 | max 0.08 | 17 – 19 | 8 – 11 | max 4% Ti | Steel stabilized with titanium |
| **Class IV – Hardened steel by ageing** | | | | | |
| 322 | max 0.07 | max 17 | max 7 | max 0.02% Al | max 0.007% Ti | |

Class III – austenite stainless steels, based on chrome-nickel, are must used in implants, especially 316 and 316L marks [19]. These steels are not hardened by heat treatments, but can be hardened by cold plastic deformation. This steels class is nonmagnetic and have a good corrosion resistance.

The austenite steels are, usually, chrome-nickel steels, which contain more than 13% chrome and more than 8% nickel. Nickel is introducing to stabilize the austenite; the addition of 2-3% molibdenum in AISI 316 and 317 steels, the corrosion resistance increase [20].

It was found that the decrease of carbon content from 0.1% to 0.03% in AISI 316 it is improved the corrosion resistance in chlorine and human physiological saline. From this reason the american standard ASTM recommend for implants to use the AISI 316L stainless steel [21].
2. Results and discussions

2.1. Spectral analysis for determination of chemical composition

From the spectral analysis effectuated on samples, using Foundry Masters Spectrometer, with iron base analysis, it is obtained the analysis bulletin present in table 1.

| Table 2. Chemical composition of analyzed alloy. |
|---|---|---|---|---|---|---|
| Fe  | C  | Si | Mn | P  | S  | Cr  | Mo |
| 52.30 | 0.12 | 1.08 | 1.46 | 0.03 | <0.01 | 24.10 | 0.12 |
| Ni  | Al | Co | Cu | Nb | Ti | V  | W  |
| 20.10 | <0.01 | 0.2 | 0.08 | 0.03 | 0.09 | 0.07 | 0.02 |

According the table 2, the studied alloy is part of class III, being considered an austenite steel, type AISI 310B, with iron, chrome and nickel as principal alloying elements.

Figure 2. Austenite domain of stainless steels [10].

Chrome is the major element form AISI 310 B steel (with content by 24.10%), fact that it offers a high corrosion resistance. Nickel, in content by 20.1%, it is used in stabilizing the austenite phase at ambient temperature and, more than that, to increase the corrosion resistance. In the case of stainless steels which contain 0.12% carbon, the stability of austenite phase can be influenced by the nickel and chrome presence (figure 2).

2.2. Structural analysis by scanning electronic microscopy

For a rigorous research in materials domain, for development and characterization of new materials or for understanding their properties it is necessary to use SEM (Scanning Electron Microscopy), in our case type Vega TESCAN LSH II

The quality of images, obtained on SEM is according by the quality of the analyzed sample and I used a detector BSE (Back Scattered Electrons).

In the images from the figure 3, obtaining by electronic microscopy, can remark details of obtained structures, for AISI 310B steel, at magnitudes between 2000- 6000 X BSE.
The AISI 310B steel presents in structure an $\sigma$ (FeCr) phase, which can cause cold deformations, but and also the presence of elements like: silicon, molybdenum, niobium, titanium. The $\sigma$ phase causes the decrease of relative elongation, of necking and resilience, increase the harness and tensile strength. In figure 3.d, can highlight in detail, carbides (points by dark color) undissolved, in a austenite base mass and presence of ferrite crystals (white color).

The influence of elements by AISI 310B steel composition on structure, is given by Schaeffler diagram (figure 4), drawing in coordinate: equivalent nickel – equivalent chrome, conforme relations:

\[
\text{Ni}_{\text{equiv.}} = \% \text{ Ni} + 30\% \text{ C} + 0,5\% \text{ Mn}
\]

\[
\text{Cr}_{\text{equiv.}} = \% \text{ Cr} + \% \text{ Mo} + 1,5\% \text{ Si} + 0,5\% \text{ Nb}
\]
By the Schaeffler diagram, result that the structure type of AISI 310B stainless steel is in function of chemical composition and the ratio of alfagene and gamagene elements.

2.3. Qualitative phase’s analysis by X-ray diffractometer investigations

Using XRD device we characterized the obtained alloys. Determination of structural constituents by diffraction is dependent to the diffracted radiation intensity and double diffraction angle.

The establishment of compositional phases was made on an X-ray diffractometer, type Panalytical X’Pert PRO MPD. It is used a fascicle by X-ray – monochrome CuKα, with nickel filter. The 2θ analysis domain is comprised between 20-80°, with a step value by 0.001°, and the step’s time is 3 second/step. Figure 5 shows the diffraction pattern obtained from the AISI 310B steel.

In sample are present like phases:
- Fe₃Ni₂ with cubic crystalline network, having a principal maximum at 2θ = 43.76° angle;
- FeCr with cubic crystalline network, having a principal maximum at 2θ = 50.58° angle.

Composition, the chemical state and structure of AISI 310B steel, highlighted by X-ray diffraction, certify the obtained results from spectral analysis and scanning electronic microscopy.

2.4. Micro-hardness measurements

Micro-hardness measurements were performed on PMT3 micro-hardness device, with standard measure of hardness HV₅₀, using a force measuring 50 g and a measurement time of 15 seconds. Were
conducted a total of 3 measurements for alloy (table 3), with the following measuring conditions: temperature 28°C (reference temperature: 23 ± 5°C) and humidity of 62%.

|     | AISI310B | N [div] | P [g] | HV   | The average value |
|-----|----------|---------|-------|------|------------------|
| 1   |          | 46,0    | 50    | 472,59 | 438,07           |
| 2   |          | 48,5    | 50    | 425,12 |                  |
| 3   |          | 49,0    | 50    | 416,49 |                  |

The orthopedic implants are required to sag, torsion, and stretch and at the contact of two pieces, due to this requests, it create tensions and in time, can appear usage. The micro-hardness, with value by 438.07 HV, obtained for AISI 310B steel, corresponding to the standard imposed by the Romanian Society of Biomaterials.

3. Conclusions

The stainless steels used in reparatory medicine cannot be eliminated, but the load to adjust these biomaterials to the integration scopes for tissues and regeneration after implant, can be a challenge for future.

The AISI 310B stainless steel is resistant to large types of corrosive agents, due to his high content in chrome (by 24.10%) and his protector oxide layer Cr₂O₃, resistant to corrosion.

The structural characterization realized by X-ray diffraction certifies the obtained results by spectral analysis and scanning electronic microscopy.

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