STUDY ON ELEMENTALS ANALYSIS PERFORMANCES OF NAA TECHNIQUES ON AISI 316L STEELS

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Abstract: *Knowing the exact chemical composition of a metallic biocompatible material is a major requirement for estimating the behavior of implant inside human body. In this direction the conformity requirements for biocompatible steels AISI 316L are specified in the SR ISO 5832-1 standard. This paper aims to provide information on capabilities of complementary techniques like NAA (Neutron Activation Analysis) for determining of a extended number of elements in AISI 316L steels.*

Keywords: *NAA (Neutron Activation Analysis), elemental analysis, biocompatible steel AISI 316L.*

1.INTRODUCTION.

AISI 316L materials are still the most used metallic biomaterials in Romania [1-3]. However they are not characterized exhaustively. To evaluate the exact properties of each alloy system is very important to understand and correlate these particulars: 1) chemical composition, 2) phase-composition, 3) crystal structure that the fine structure of the polymorphic phase microstructure, 4) distribution and preferential orientation grain (texture), 5) synergistic effects on mechanical properties, electrical and biocompatibility. This scientific way of characterization of biomaterials is required, but is very expensive and therefore must find compromise solutions between the level of knowledge and cost [1-3].

Alloy 316 L contains predominantly iron (60-65%) alloyed with large amounts of chromium (17-19%) and nickel (12-14%) and minor amounts of nitrogen, manganese, molybdenum, phosphorus, silicon and sulfur. The reason to adding these alloying elements is to finishing the microstructure of metal. The key role of chromium is to allow development of a surface layer resistant to corrosion by forming a strong adherent oxide to the surface (Cr_2O_3). Molybdenum and silicon are ferrite stabilizers. To counteract this action of forming ferrite, nickel is added to stabilize the austenitic phase.

This steel is less than 0.030% carbon for to reduce the susceptibility to corrosion in the body. When the carbon content of steel significantly exceeds the value of 0.030%, then there is danger to formation the carbides. Chromium carbide precipitation in adiancent regions impoverishes the grain boundary, which has the effect of reducing the capacity of forming the protective layer of Cr_2O_3. Steels in that form carbides are also sensitized, vulnerable and tend to give the accidental breaks. They are favored by extremely aggressive corrosion in the body that creates primers grain breaking limit.

In accordance with ASTM specifications, structure 316 L is the desired single phase, austenite (face centered cubic). The metal should be free ferrite (volume centered cubic) or phases of carbides in the microstructure. Also, the steel must not present the inclusions such as sulfides. Grain size suitable for 316 L is ASTM 6 or finer.

Of those mentioned above on the requirements needed to satisfy type 316L steels used as biomaterials results that accurate spectral analysis phase is required in obtaining them. This phase is extremely critical, because the measurements must be performed while steel is melted in the oven, expecting approval of its composition to be casting. Certification composition is corrected depending on the structure of cast samples must be done in about 10 min to solidify the evidence. Otherwise, change the composition of the furnace due to combustion of easily fusible alloy elements (Al, Mg, etc.) and the charge is rejecting, for example lost about 700 €. Spectrometric analysis performed on AISI 316L after further treatment or processing, must take into account the fact that their structure may influence the accuracy of chemical analysis [4,5]. 316 L steels are in the form of metal sheets, strip, plate, ASTM A240 standard.

These materials have special expertise conditions. Because these materials come into direct contact with living tissues, in addition to low toxicity, should not create adverse reactions in humans. Due to special requirements imposed by the use of 316L steel in orthopedics, shall be determined with precision of 0.005% of the elements C, S, P. This, in terms spectrometry is a very difficult task, which is situated at the highest level of performance in the field [6].

Outdoor steel melting 316L can lead to his contamination and consequently to the low biocompatibility characteristics. For this reason prefer 316LVM steel, low carbon steel, melted in a vacuum. A steel 316L or 316LVM is considered biocompatible if it meets ASTM F 138, or ISO 5832-1 [6].

Table 1. The chemical composition of steel 316 L, comparative presentation standards AISI, ASTM, ISO.

Element	AISI max%	ASTM max%	ISO max%
C	0,03	0,03	0,03
Mn	2,0	2,0	2,0
P	0,045	0,025	0,025
S	0,03	0,01	0,01
Silicon	0,75	0,75	1,0
Cr	16,0-18,0	17,0-19,0	17,0-19,0
Ni	10,0-13,0	14,0-15,0	14,0-15,0
Mo	2,0-3,0	2,25-3,0	2,25-3,5
N	0,1	0,1	0,1
Co	nu se impune	0,5	0,5
Fe	restul		

2. EXPERIMENTAL DETAILS.

In this work we aimed to expand the number of elements as much dosage of AISI 316L alloys, using conventional nuclear and atomic methods. However, nuclear techniques including neutron activation analysis (NAA) have been scarcely used in the analysis of such materials. This fact can be explained by the difficulty of access to nuclear facilities and the great development, in the last few years, of less expensive techniques like ICP, AAS and XRF.

Instrumental neutron activation analysis was applied to evaluate the chemical composition of metallic materials, Accuracy and precision results of about 10% were achieved for most elements, indicating that the technique is suitable for the analysis of metallic materials.

Full elemental analysis, ie determination of all elements of Mendeleev's table is an enterprise scale technology, but also economical, which can be implemented with common means. Therefore, at present limited number of items dispensed, if that type AISI 316L biomaterials, according to SR EN 13005 / 2003, ISO 5832-1 and the factors which determine are C,, Mn, P, S, N, Cr, Mo, Ni, Cu, Fe [7].

Given the possibility NAA analytical techniques to dose order elements are in ppm concentration was considered useful to extend the investigation of steels type AISI 316L compositional possibilities beyond SEOASE analytical technique. Biocompatible metallic materials of AISI 316L grade materials are most representative for achieving implantable prostheses. For this reason, these materials were chosen to test the performance of analytical methods, and to estimate what extra can bring an advanced nuclear analytical method and NAA method.

To ensure traceability of analytical tests NAA and SEOASE using a reference sample of type AISI 316L whose composition was determined in a network involving more intercomparable analytical laboratories. Reference sample composition and compositions determined by NAA and SEOASE techniques on samples taken from reference samples are presented in Table 2 [7]

Table 2.Assigned composition and test and NAA performed SEOASE reference sample of steel AISI 316L [7].

Element	B	C	N	Na	Al	Si	P	S	K	Ca	Ti	V	Cr	Mn	Fe
	ppm	%	%	ppm	%	%	ppm	ppm	ppm	ppm	%	%	%	%	%
C_A	20	0,027	0,08	41	0,08	0,63	170	20	41	10	0,19	0,09	17,9	1,91	63.3
Cimp		<0,03	<0,1			<1,0	<250	<100					17-19	<2,0	
SEOSE	22	0,028	0,1		0,083	0,64	181	22		13	0,17	0,11	17,6	1,83	63,7
AAN	17	0,021	0,07	34	0,076				47	12		0,13	18,01	1,91	
ARs(%)	10,0	3,7	25,0		3,8	1,6	6,5	10,0		30,0	10,5	22,2	1,7	4,2	0,2
AR_{AAN}(%)	**15,0**	**22,2**	**12,5**	**17,1**	**5,0**				**14,6**	**20,0**		**44,4**	**0,6**	**0,0**	

Table 2 below

Element	Co	Ni	Cu	As	Nb	Mo	Sb	La	W	Au	Pb
	%	%	%	ppm	%	%	ppm	ppm	%	ppm	ppm
C_A	0,12	13,05	0,03	21	0,04	2,3	49	51	0,27	28	14
Cimp		13-15	<0,5			2,25-3,5					
SEOSE	0,15	13,01	0,03		0,05	2,26			0,24		
AAN	0,11	12,93	0,04						0,32	28	14
ARs(%)	25,0	0,3	0,0		25,0	1,7			11,11		
AR_{AAN}(%)	8,3	0,9	33,3						18,5	0,0	0,0

3. RESULTS AND COMMENTS.

Comparative analysis of analytical data obtained by techniques SEOASE and NAA results that elements have a concentrations of order of percentage differences between the two techniques are <7%. In these cases the techniques have comparable performance. If elements with concentrations <0.1% and especially at the ppm (trace), comparable analytical results differ by more than 15%. Apparently the two techniques provide inconsistent results.

On the other hand, as was shown in work [8] at low concentrations, ppm sites, the relative uncertainties are> 50%. Thus, for each technique in some special measures must be taken in order to ensure the quality results. In this case there was no question the quality of experimental results and inter-comparison results. But in terms of cost and rapidity of analysis is obviously SEOASE technique is superior. Also, methods are laborious and nuclear techniques both in terms of complexity of mathematical modeling and measuring uncertainty of the budget consists of physical factors such as particle flow fluctuations, fluctuations of the chain of detection, and scattering effects absorption area, (other than the sample), etc. Also, NAA technique presents these undesirable aspects such as the fact that for some radioisotopes, the time elapsed from irradiation to the end of the analysis of samples is too large, and some elements present in steels and a great influence on their properties, such as S, P, Ti, Si, etc., can not be analyzed because of very short half-life of radioactive nuclei or neutron capture sections too small [9]. Thus, the dosage related conformity assessment matrix composition of metal is obvious that we recommend using the system of accreditation tests SEOASE RENAR.

In conclusion NAA technique is recommended for more accurate analysis of chemical composition, at ppm level, for the metallic materials.

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