EXPERIMENTAL CHARACTERIZATION OF TWO STAINLESS STEEL EXPLANTED ENDER PINS

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În urma explantării, au fost analizate două tije Ender din punct de vedere al compoziţiei chimice, au fost caracterizate structural şi au fost determinate proprietăţile mecanice şi de rezistenţă la coroziune. Analiza comparativă a celor două tije cât şi datele experimentale au referinţe directe cu cerinţele standardelor ASTM şi ISO pentru implanturi. Având în vedere faptul că aceste tije au fost în serviciu timp de aproximativ şapte luni, rezultatele experimentale permit o estimare a evoluţiei în timp a proprietăţilor materialului cât şi informaţii valoroase pentru a observa performanţa materialului.

This paper presents the comparison of two explanted Ender pins. The pins were studied regarding their chemical composition, their structure, mechanical properties and corrosion resistance. The tests and the evaluation of results were according to ASTM and ISO standards. An important aspect is that both pins had a work life of about seven months. Thus the experimental results are useful to evaluate the material properties in time and also to deliver important data for material performance.

Keywords: Ender pin, intramedullary fixation, corrosion, mechanical properties

1. Introduction

The human bone is a living tissue suffering a continuous transformation. Its complex structure causes a large variety of properties. The bone is the most resistant piece to deformation in the human body. It is not a homogenous material; therefore the mechanical properties vary regarding coordinates [1]. Intramedullary fixation surpasses the bone fixation plate's method, when speaking of bones subjected to mechanical stress. The Ender pins are inserted without bone drilling and could be used as a bundle. They can be inserted in the long bones of the human body. [2]

These pins have small diameters compared to the intramedullary canal, a uniform curvature and variable lengths. [3]

The materials of which the Ender pins are made of are usually stainless steels. In this paper of have made a thorough characterization of Ender pins made of an austenitic stainless steel after explantation.

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The analyzed pins were obtained after explantation at equal time intervals, from different patients.

For easy identification the pins were conventionally noted as follows: the first pin was noted E1, the second one E2.

2. Results and Discussion

2.1. Spectrometric analysis

The chemical composition of the investigated pins is given in following tables as obtained by optical spectrometry analysis. The chemical composition was compared with ASTM and ISO standards specifications.

The experimental results and the comparison are presented in table 2.1.1.

To analyze the future behavior of the Ender pins in service we have investigated the content of inclusions, the micro hardness and the corrosion resistance [7]. The chemical composition was determined using the optical spark emission spectrometer SPECTROMAXx.

Using the obtained results the steel was identified and compared with the standard specifications and several theoretical coefficients were calculated.

Table 2.1.1

Chemical compositions for investigated stainless steels compared to ASTM F55-66 and F56-66 and ISO 5832-1:1997(E) specifications

Element	Chemical composition ASTM (%)	Chemical composition limits (%)		Composition limits according to ISO 5832-1:1997(E)[%]		Investigated samples chemical composition (%)	
		316	316L	Composition D	Composition E	E1	E2
C	max.0.08	max.0.10	max.0.03	max.0.030	max.0.030	0.026	0.024
Si	max.0.75	max.1.0	max.1.00	max.0.030	max.0.030	0.026	0.024
Mn	max.2.0	max.2.0	max.2.0	max.2.0	max.2.0	2.19	1.88
P	max.0.03	max.0.04	max.0.025	max.0.025	max.0.025	0.022	0.019
S	max.0.03	max.0.03	max.0.015	max.0.010	max.0.010	0.0051	0.0051
N	n/s	n/s	n/s	max.0.10	0.10-0.20	0.057	0.075
Cr	17-20	16-18	16-18	17.0-19.0	17.0-19.0	18.76	19.23
Mo	2.0-4.0	2.0-3.0	2.0-3.0	2.25-3.5	2.35-4.2	2.39	2.6
Ni	10-14	10-14	10-14	13.0-15.0	14.0-16.0	12.71	13.13
Cu	n/s	n/s	n/s	max.0.50	max.0.50	0.076	0.049

The obtained chemical compositions for the two samples were compared with ISO 5832-1:1997(E) specifications. Both pins conformed to standard requirements under composition D. The high chromium content corresponding to pin E2 exceeds the highest limit value from ASTM F55-66 and F56-66 by circa

1.23%, thus failing to conform to standard specifications. In the same situation is pin E1 which exceeds the superior limit prescribed for manganese by 0.19% and that for chromium by 0.76%. According to the chemical composition the steels were classified according to table 2.1.3 and the „C" coefficient was computed according to the following formula:

$$C = 3.3 \times \%Mo + \%Cr \quad (1)$$

The chemical compositions were compared with AISI and DIN to find out the type of steel used.

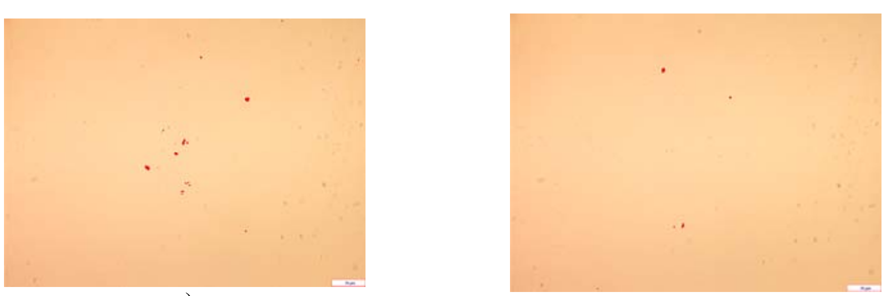
Table 2.1.2

The calculated values using the chemical composition

Pin	Steel		According ISO 5832-1:1997(E)	C	According SR ISO 5832/97
	AISI	DIN			
E1	316L	1.4435	Yes	26,65	C>26, Yes
E2	316L(medical)	1.4441	Yes	27,81	C>26, Yes

2.2. The computerized qualitative and quantitative analysis for inclusions and microstructure

The qualitative and quantitative analysis was performed, as required by ISO 5832-1:1997(E), on longitudinal and transverse cross-sections, meaning the parallel and perpendicular directions reporting to the deformation direction. The characterization regarding structural discontinuities was performed by determining at least three fields, their percent and the occupied surface and their sphericity coefficient. The computerized analysis line consists of a UnivaR Reichert microscope and three modules analysis software produced by Media Cybernetics, USA, following the standard specifications from ASTM E1245. The studies were performed on three different fields. The analyzed surface consists of an area of 432471 μm^2 and it was maintained constant. After determination the structural discontinuities from the selected fields were counted.



a) b)

Fig. 2.2.1 Captured and processed image for pin E1 on a)longitudinal and b) transverse cross-section

a)

b)

Fig. 2.2.2 Captured and processed image for pin E2 on a)longitudinal and b) transverse cross-section

Table 2.2.1

Field area percent for inclusions in pins E1 and E2 on longitudinal and transverse cross-sections

Sample	Cross-section	Max[%]	Min[%]	Mean[%]
E1	Longitudinal	0.0729687	0.0321354	0.0475521
	Transverse	0.0408854	0.0238583	0.0300694
E2	Longitudinal	0.0135417	0.00536458	0.00996528
	Transverse	0.0411979	0.0230729	0.0331424

Table 2.2.2

Feature area for inclusions in pins E1 and E2 on longitudinal and transverse cross-sections

Sample	Cross-section	Max[μm^2]	Min[μm^2]	Mean[μm^2]
E1	Longitudinal	69.1503	4.95539	14.3476
	Transverse	92.1253	4.95539	22.9485
E2	Longitudinal	20.4973	4.73015	11.7537
	Transverse	56.5365	6.08162	19.5451

Table 2.2.3

Feature sphericity for inclusions in pins E1 and E2 on longitudinal and transverse cross-sections

Sample	Cross-section	Max	Min	Mean
E1	Longitudinal	0.827936	0.256375	0.625511
	Transverse	0.820214	0.509884	0.664181
E2	Longitudinal	0.815286	0.550251	0.68947
	Transverse	0.803117	0.37964	0.651513

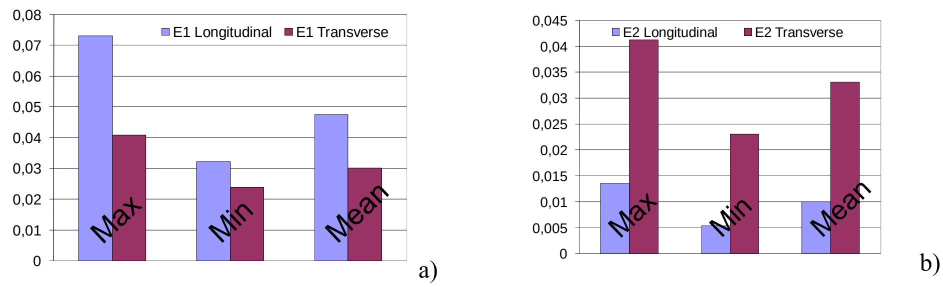


Fig. 2.2.3 Histograms indicating the occupied percent of the investigated surface by structural discontinuities and non metallic inclusions on transverse and longitudinal cross-sections for a) pin E1 and b) pin E2

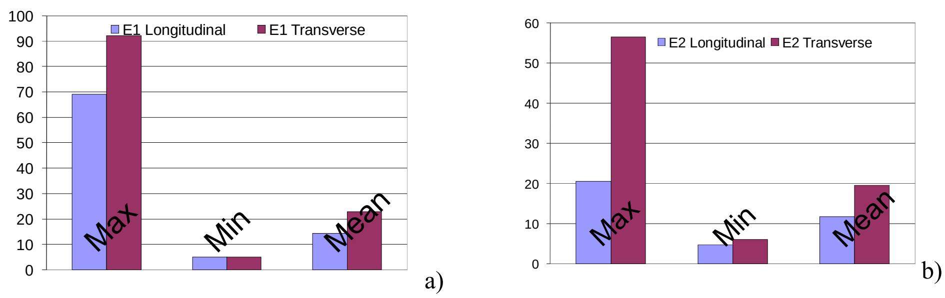


Fig. 2.2.4 Histograms indicating determined areas for structural discontinuities and non metallic discontinuities on longitudinal and transverse cross-sections for a) pin E1 and b) pin E2

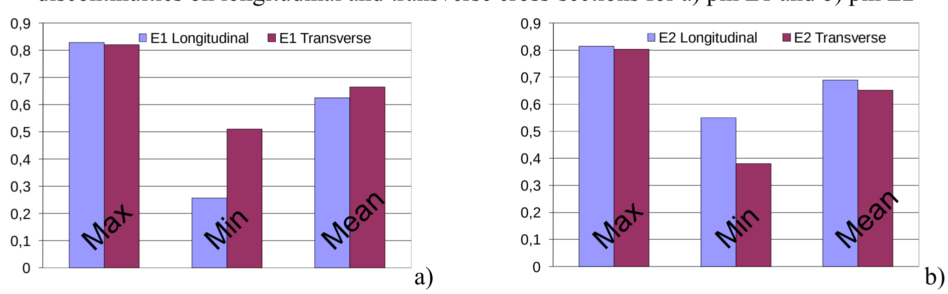
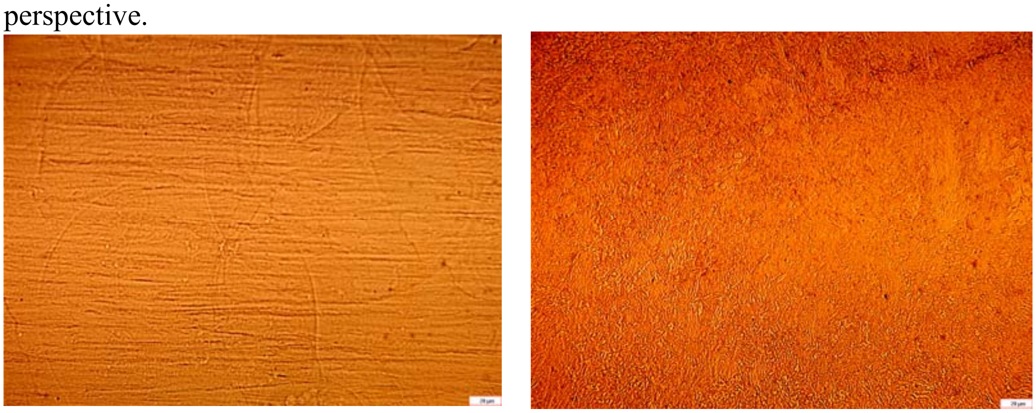


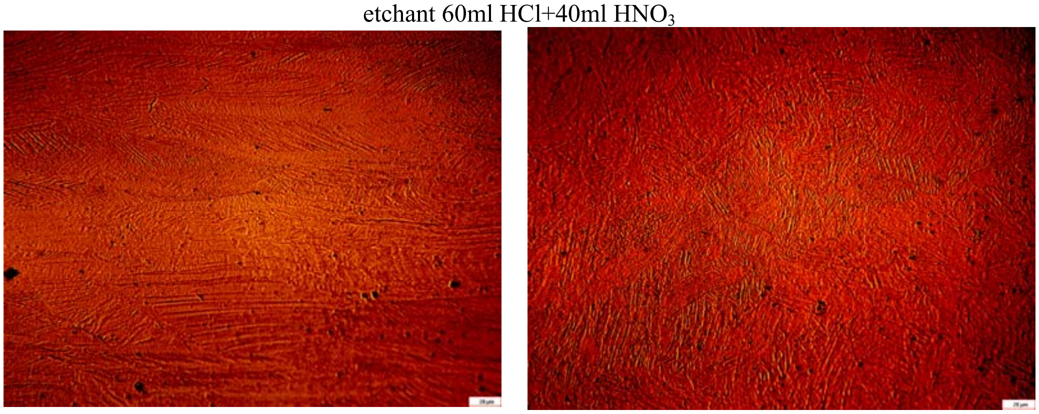
Fig. 2.2.5 Histograms indicating sphericity coefficients for structural discontinuities and non metallic inclusions on longitudinal and transverse cross-sections for a) pin E1 and b) pin E2

Comparing the obtained values it is obvious that pin E1 presented larger values for the maximum, minimum and the mean of the field area percent on longitudinal cross-section. The differences calculated were 0.059427% for the maximum value of the field area percent, 0.026771% for the minimum and 0.037587% for the mean value. On the transverse cross-section the minimum value for the field area percent of pin E1 exceeds the value of E2 by 0.000785%. Regarding the maximum and the mean values for pin E2 it is noticed that these values are exceeding the ones of pin E1, but barely noticeable. By observing the feature area percent on longitudinal cross-section it was concluded that pin E1 shows larger values for the maximum, the minimum as well for the mean. On transverse cross-section pin E2 presents a larger value on the minimum, by 1.12623 μm^2, the maximum and the mean values being exceeded by the ones corresponding o pin E1. On the feature sphericity for the inclusions on longitudinal cross-sections pin E1 shows a larger value for the minimum, the maximum and the mean values are exceeded by the values of pin E2. On transverse cross-sections the values for the minimum, maximum and the mean for pin E1 are exceeding the ones for pin E2. The values do not differ significantly for the maximum and the mean, but, regarding the minimum values, the difference between values for pin E1 and values for pin E2 is 0.130244. Comparing the values and observing the graphs it was concluded that the material which constitutes pin E2 presents a better homogeneity and should perform better in all the aspects which might imply the discontinuities and non metallic inclusions perspective.



a) b)

Fig. 2.2.6 Optical micrographs of pin E1 on a) longitudinal and b) transverse cross-section, 500X, etchant 60ml HCl+40ml HNO_3



a) b)

Fig. 2.2.7 Optical micrographs for pin E1 on a) longitudinal and b) transverse cross-section, 500X, etchant: 60ml HCl+40ml HNO_3

Observing the optical micrographs it is noticed that sample E1 is cold worked, with a large deformation degree, the microstructure thus obtained being typically fibrous.

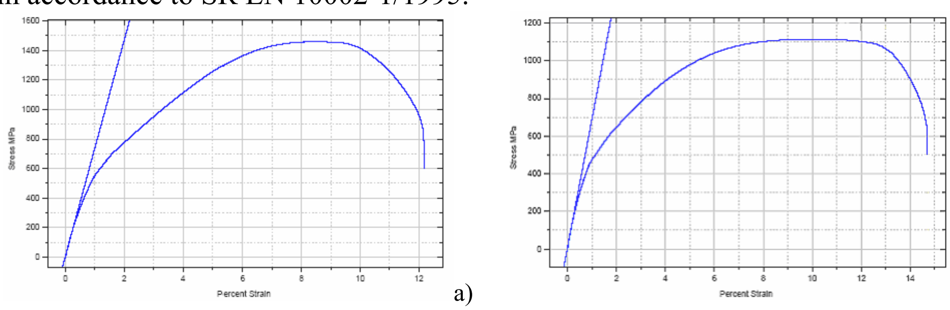
The optical micrograph of test sample E2 on longitudinal cross-section shows an austenitic structure, with twins, as a result of cold working, the grains following the deformation direction applied.

Appreciating the aspects of the micrographs it was concluded that both pins were subjected to cold working.

Pin E1 had a larger deformation degree than pin E2 [4]. Although the chemical attack could have highlighted other constituents, none was observed in the analysis of the microstructure.

2.3 Mechanical resistance

To determine the mechanical resistance, a universal testing machine INSTRON 3382, with 10kN maximum force was used. The tests were performed in accordance to SR EN 10002-1/1995.



a) b)

Fig. 2.3.1 Tensile stress curves for a) pin E1 and b) pin E2

Table 2.3.1

Comparison for the values obtained by mechanical tests

Sample	D_0 [mm]	A_{gt} [%]	A_t [%]	Rm [N/mm^2]	Z [%]
E1	4.42	8.51	12.20	1459.7	63.78
E2	4.76	10.08	14.70	1114	63.90

Analyzing the values it was noticed that the test sample E1 had a mechanical resistance greater than E2, but the striction and total elongation values were smaller.

Regarding the elongation until failure both Ender pins conform to ISO 5832-1:1997(E) specification, the minimal prescribed value is 12%, pin E1 exceeding this value by 0.20% and pin E2 with 2.70%.

According to ISO 5832-1:1997(E) the superior limit value for the mechanical resistance is 1100 MPa, thus, both pin E1 and E2 exceeding this value by 359.7 MPa and 14 MPa.

It could be concluded that pin E1 does not satisfy standard specifications, as for pin E2 the value exceeds the standard specification by 1.27%, an acceptable value, thus pin E2 conforming to the standard specification.

2.4 Micro hardness tests

The measurements were performed on a Karl Zeiss Jena microscope equipped with a Hanneman indentor with a 40g load and 20 seconds maintaining interval.

Three tests were performed on each sample to determine the Vickers micro hardness, on longitudinal and transverse surfaces.

Table 2.4.1

Micro hardness values

Sample	Cross-section	Test 1 [$\mu HV_{0.40}$]	Test 2 [$\mu HV_{0.40}$]	Test 3 [$\mu HV_{0.40}$]	Mean [$\mu HV_{0.40}$]
E1	Longitudinal	477	418	467	454
	Transverse	525	566	594	562
E2	Longitudinal	400	400	418	406
	Transverse	409	418	467	431

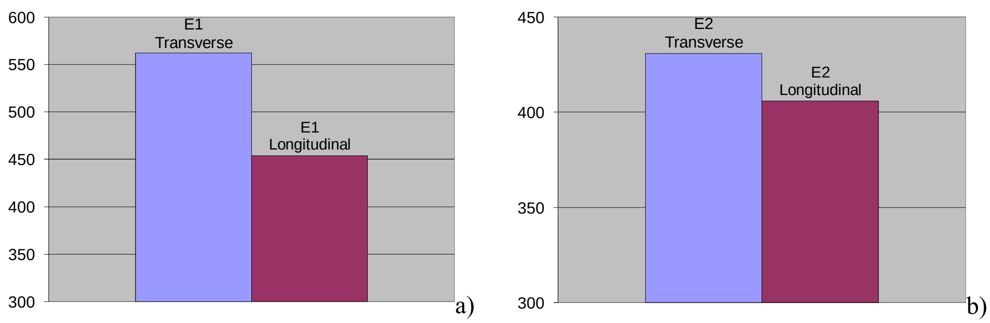


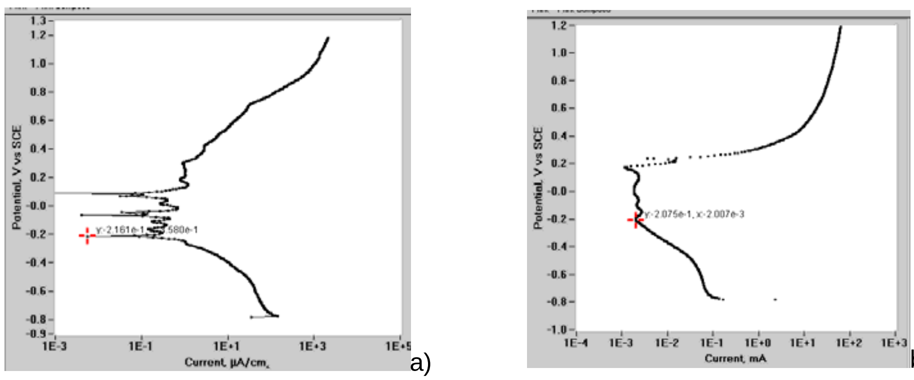
Fig. 2.4.1 Histogram indicating the average micro hardness values on longitudinal and transverse cross-section for a) pin E1 and b) pin E2

On longitudinal cross-section the difference between the averages values of the micro hardness for pin E1 and pin E2 is 48$\mu HV_{0.40}$ and on transverse cross-section 131$\mu HV_{0.40}$.

2.5. The corrosion results

The corrosion tests were performed on a Potentiostat/Galvanostat Model 2049 apparatus which plots the cathodic curves according to ASTM G5, equipped with a function generator model 586 and acquisition board connected to a personal computer.

The test samples were prepared by sanding and polishing, like the metallographic preparation. The analyzed surface was transversal, parallel to the deformation direction. The test samples were immersed in 500ml physiological solution with the following chemical composition: 4.22 g NaCl, 0.175g $NaHCO_3$ and 0.03 g NaH_2PO_4. The measured pH value was 6.82.



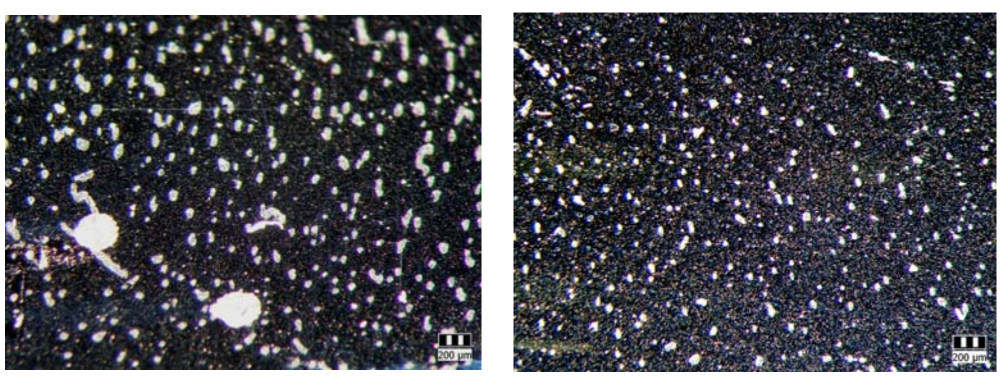
a) b)

Fig. 2.5.1 Potentiodynamic curves for a)pin E1 and b)pin E2

Analyzing the potentiodynamic curves it was noticed that test sample E2 shows a better corrosion resistance than E1.

2.6. Macroscopic analysis

The macroscopic analysis was performed on the stereomicroscope Olympus SZX7. The samples were analyzed using the stereomicroscope before and after the corrosion tests.



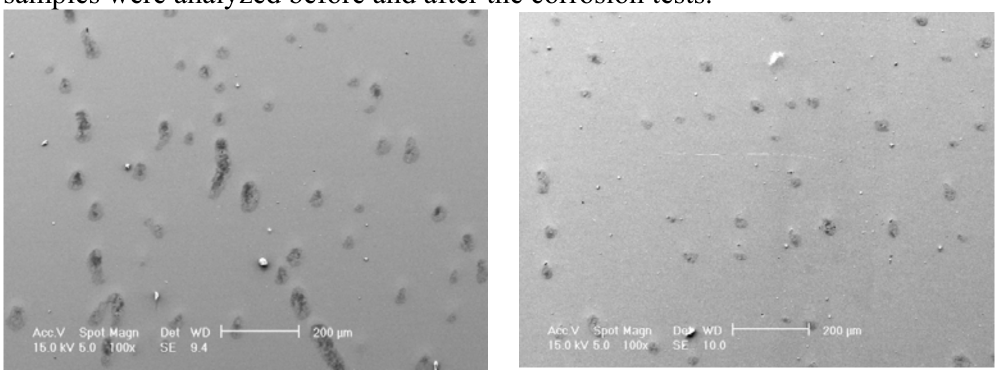
a) b)

Fig. 2.6.1 Pins a) E1 and b)E2 after corrosion tests

After stereomicroscopic inspection no corrosion aspects were noted on both Ender pins. After corrosion tests the samples were analyzed using stereomicroscopy technique, observing the reaction products on the surface of both samples.

2.7 SEM analysis

The microscope used was a XL 30 ESEM TMP FEI-Philips. The test samples were analyzed before and after the corrosion tests.



a) b)

Fig. 2.7.1 Corroded pins a)E1 and b)E2, image using secondary electrons detector, 250X

Analyzing the exposed surfaces spotted corrosion aspects were observed on both pins, mentioning that the distribution and size of the spots in the case of pin E1 exceeds the ones in case of pin E2.

3. Conclusions

Using spectrometric techniques to determine the chemical composition of the pins, the steels were classified as 316L for pin E1 and 316L (medical) for pin E2. Both pins successfully conformed to ISO 5832-1:1997(E) under composition D but failed to conform to ASTM F55-66 and F56-66 because of high contents of manganese and chromium.

The pins E1 and E2 conformed to standard specifications regarding the value of coefficient C, the calculated values exceeding the minimal specified value 26.

Analyzing the inclusionary states by feature area, field area percent and by sphericity coefficient we concluded that both pins presented a good inclusionary state, making them adequate for their intended use.

The alloy which constitutes pin E2 presents a better homogeneity than E1, and both pins should perform better in all the aspects which might imply the discontinuities and the non metallic inclusions perspective.

Analyzing the microstructure of the test samples on longitudinal and transverse cross-section it was observed that pin E1 showed a typical austenitic structure with a large deformation degree, while pin E2 showed an austenitic structure with a lower deformation degree.

No other constituents were observed during the microstructure investigation, thus conforming to ISO 5832-1:1997(E) requirements.

Appreciating the aspects of the micrographs it was concluded that both pins were subjected to cold working. Pin E1 had a larger deformation degree than pin E2 [4].

From the mechanical tests performed it was noticed that test probe E1 had a mechanical resistance greater than E2, but the striction and total elongation values were smaller.

Both pins conform to ISO 5832-1:1997(E) requirements regarding their minimal elongation until failure, but pin E1 fails to conform to ISO 5832-1:1997(E) because of its mechanical resistance value which exceeds the superior limit prescribed in the standard by 32.7%.

Pin E2 conforms to the standard specifications for the mechanical resistance and elongation [6].

The micro hardness tests show larger values in case of pin E1 on longitudinal as well on transverse cross-section.

Examining the pins before the corrosion tests no corrosion aspects were visible. Immersing the samples in a biological similar fluid, an artificial serum, both pins suffered a corrosive attack.

The distribution of the peaks and the general aspect of the potentiodynamic curves tend to express that pin E2 presents a better corrosion resistance in the selected environment [5].

During stereomicroscopic analysis no corrosion aspects were noted on the pin. When performing the corrosion tests and analyzing the samples through stereomicroscopic technique deposits of the reaction products were observed on surfaces of both pins.

In the SEM analysis the exposed surfaces during the corrosion tests presented spotted corrosion aspects on both pins [8]. The distribution and the size of the spots on sample E1 exceed the ones corresponding to sample E2.

Both pins had a good performance during all tests. Keeping in mind that they were used for 7 months in service in a very aggressive environment, the human body, both pins maintain excellent mechanical properties and a very good corrosion resistance.

Despite the different alloys which constitute the pins, both performed excellent in their service life, after investigations no obvious aspects of failure were noticed.

By comparing the obtained data and performing a thorough analysis of each alloy properties and behavior it is clearly noticeable that the one which constitutes the pin noted E2 shows better altogether values, thus pin E2 should have a better performance in service.

R E F F E R E N C E S

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