The study of the mechanism interaction between sparks electric discharges and a AISI 316L biocompatible metallic samples

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Abstract. This article is a study of the mechanism interaction between sparks electric discharges and a AISI 316L biocompatible metallic samples. The AISI 316L steel samples were analyzed compositionally by OES spectrometry and the sparked areas in the samples were subjected to optical microscopy and SEM electron microscopy investigations. The experimental results highlighted new aspects regarding the attack mode of sparks with the metal sample and explain why the edges of the sparked areas are blackened / smoked.

1. Introduction
Compositional (elemental) analysis of conventional metal materials (base alloys Fe, Al, Cu, Co, Ni etc.) is performed almost exclusively by OES spectrometry [3,5,9,11,12]. Compositional analysis of special metallic materials such as biocompatible alloys, aviation alloys, nuclear reactor alloys, etc., involves special metering methods and techniques, which ensure the quality of the test results, according to the requirements regarding the estimation of their conformity with the related requirements (e.g. Law 608/2001 and SR EN 573-3 / 2009 [2,13] for aluminum alloys for aviation) and Law 608/2001 in conjunction with SR ISO 5832-1 and the standards SR EN 17025 and SR EN 13005 [6,7]. A category of special materials is represented by biocompatible steels for orthopedic applications, respectively AISI 316 L steels, for which research has been done at SC COST SA Targoviste, Valahia University Targoviste and Politehnica University of Bucharest. For these types of materials, the problem of compositional analysis with high accuracy was raised for validating their application in medical practice [2,14]. OES spectrometry (optical emission spectrometry) by electric spark) represents the most effective technique of compositional analysis of metal alloys. In fact, more or less intentionally, the elemental analysis of the special metal alloys is performed by OES spectrometry, either for preliminary or routine tests, in order to identify the alloy class (e.g. Dural type 2017 or 2024), or even for assessing the conformity of the alloy composition with the related specification. In the case of the spectrochemical tests for the evaluation of the compositional conformity, special measures must be taken to ensure an adequate accuracy of the test results, which is not quite within the reach of the testers without adequate studies.

Also, regarding the spark electric discharge used in OES spectrometry, it is considered that it would have a temperature of about 30 000 K, which would allow instantaneous vaporization of the material from the discharge area. Also, it is known that the spark electric discharge is started on inclusions from the metal sample, consisting of slag particles, compounds or abrasive particles from
the "sanding" paper, e.g. corundum. For this reason, "soft" materials (Al alloys, Pb alloys, Sn alloys, etc.) cannot be sanded with abrasive paper (Al₂O₃ or SiC) because they will be impure. These samples are processed by turning, milling, etc.

2. Techniques and methods.
The samples tested by OES spectrometry are two types obtained in SC COST Targoviste and a reference sample provided by the LISEOFRX Laboratory of UPB-SIM. Because AISI 316L biocompatible steels are topical in their importance for medical implantology, samples from this category have been investigated because it was followed that:

1. elucidation of some aspects that underpin the spark theory,
2. obtaining the data necessary to improve the spectrochemical testing of these steels, which are part of the products notified by Law 608: 2001, as materials whose conformity has to be certified according to the standard SR EN ISO 7153-1: 2003 [16].

Thus, in the case of a steel sample (eg AISI 316L) it is estimated that during a spark, consisting of all the sparks produced during the measurement of the "intensity" of the spectral line, about 10⁻⁴ grams of the substance sample is vaporized (e.g. Fe), this mass is made up of about 10¹⁶ Fe atoms [1,4]. From the specialized works [8, 10,15,17,18] it is concluded that vaporization and excitation of UV-VIZ emission are two coupled factors, which in turn depend on both the nature and state of the sample, as well as the electrical parameters of the discharge, which are:

1. The size and shape of the discharge gap;
2. The power dissipated during a discharge (spark);
3. The frequency of discharges by spark or alternating electric arc;
4. The volume of vaporized material during a discharge;
5. By the distribution of the individual discharges, respectively of the craters of discharge on the surface of the sample (of the discharge spot);
6. By the number of atoms that undergo ionization in the discharge space and emit ionic lines;
7. By the number of excited atoms emitting characteristic atomic lines, respectively the analysis line.

In order to have clear images regarding the electric discharge by spark, the sparked areas, by optical microscopy and SEM-EDS electron microscopy were investigated. Macroscopic images of the sparked areas are shown in figure 1. As shown in figure 1 the sparks have a specific appearance. There is a variation in gray levels from the center (almost white) to the edge (almost black). In the common sense, the edge of the sparkling area seems to be blackened, smoked. To elucidate this fact, microscopy analyzes were performed as will be shown below.

![Figure 1](image_url)

The microstructures were visualized with a computer-assisted REICHERT UnivaR microscope equipped with image analysis software. The device is equipped with a high resolution digital camera type Polaroid DMC 1E type TWAIN driver. The image analysis equipment has a Frame Grabber type Matrox Meteor II. The optical microscopy investigations do not provide sufficiently accuracy to elucidate the mechanisms of interaction of sparks with the AISI 316 L steel samples. Therefore, it was considered that a SEM (scanning electron microscopy) investigation would be superior to optical microscopy investigations. The XL-30-ESEM TMP electron microscope equipped with EDS
spectrometer was used for the SEM investigation of the scanned areas. The microscope is equipped with software suitable for operating assistance, data acquisition and results processing. The microscope provides electron acceleration voltages in the range of 5-45 keV, magnifications of 5-105x and all the facilities specific to an SEM microscope.

3. Experimental results.
To obtain information on the interaction of the spark discharge used as a spectral source with the AISI 316L steel sample, four representative sparking areas were investigated. Each sparked area was visually examined after which at least one image from the morphologically differentiated areas was acquired, respectively overall images, images from the center of the sparked area, images from the median area and images from the peripheral area of the sparked area. Following the analysis of the images purchased for each sparked area, the most representative images were selected, which are presented below. For the investigation of the scanned areas, images were used for atypical magnifications denoted 1X, 1X bis and 4X whose magnification scales are presented in figure 2.

![Magnification scale used in microscopic investigation.](image)

3.1. Microscopic investigation of the sparked area no. 1.
The image in figure 3 shows the overall appearance of the sparking surface. From the granular aspect of the central area, which differs from the rest of the surrounding surface, the average diameter of the spark can be estimated. But this is not relevant in terms of accuracy, because there is a transition zone from the scanned area to the surrounding area, which has a diffuse appearance, with nonhomogeneous distribution of the incidence of sparks. The image shows aspects that suggest the existence of pores or pits in the sparking area. It also figure 3 demonstrates, without denial, that the sparking area does not have a uniform morphological appearance.

![Overview of the sparked area no. 1](image)

![Image from the central area of the sparked area no. 1](image)

![Image from the edge area of the sparked surface no. 1](image)

In figure 4 are presented the morphological aspects of the sparking area (spark 1). And this image reveals a locally nonhomogeneous granular morphology, but homogeneous at the zone level, respectively the granular aspect of the central area is homogeneous in relief, at the scale of tenths of a
millimetre. The image from the transition area figure 5 shows a rougher morphology than the one from
the central area, which can be corroborated with the aspects revealed by figure 3.

The most important aspect, revealed by the metallographic optical microscopy, is the coarser
(more cracked) granular aspect of the center of the sparked area, in relation to the adjacent areas. On
the other hand, the observation of the sparks with the naked eye suggests that the marginal areas of
the sparked area are rougher. In fact, the "smoky" appearance of the spark crown suggests this effect.

3.2. Microscopic investigation of the sparked area no. 2
The overall image of the sparked area no.2 (figure 6) shows, without any doubt, that the sparked area
is bordered by a crown of pitting whose existence can only be justified by concentrating the spark on
inclusions. Following the massive attack, the inclusions are vaporized or eroded (etching / sputtered)
leaving behind pores / holes / pitting.

In the circle delimited by pores (figure 6 b) there are other nonhomogeneous distributed pores,
mainly in quadrants I and IV of the circle. Above the circle are a few smaller pores. The image in
figure 6 clearly proves that an analysis based on a spark can be completely erroneous, because the
analytical signal is not proportional to the concentration of the elements in the sample, but is distorted
i.e. increased for items in inclusions. The images in figure 6 indicate that the selective attack is made
at quasi-vertical incidence, because the edges of the pores are not excavated, i.e. the pores have quasi-
cylindrical morphologies that do not degenerate into cones.

3.3. Microscopic investigation of the sparked area no. 3
Spark no. 3 presents morphological details similar to spark no. 2, which shows the stability of the
phenomenon. In this case, two overviews are presented, because they complement each other
extraordinarily well. Figure 9 clearly shows the morphology of the sparked zone, but especially the
zone to the non-sparked zone, i.e. there is an attack gradient morphology of the transition zone from
the sparked.
If we analyze together figure 9 and figure 10 it is observed that the morphology of the sparked area has a well-defined coloristic aspect, respectively the central area of the sparked area is lighter than the crown dotted with pits, which in turn has a darker color than unscented area. Figure 9 better reveals this aspect, as well as the risks specific to the correct processing of the sample. The image of the central area in figure 11 reveal a granular morphology with more pronounced micro-excavations and bright areas. Figure 11 reveal a more pronounced attack in certain areas (black ones) and (sparks incidence) lower in brighter areas. The image in figure 12 reveals the morphology of a marginal area, but within the perimeter of incidence of sparks. Basically, on figure 12 more prominent attacks can be located. Thus figure 12 brings morphological arguments in favor of an nonhomogeneous attack of the sample in the spark process.

3.4. Microscopic investigation of the sparked area no. 4

Figure 13 reveals a different mode of attack than the one in the previous figures, respectively an attack with pitting in the centre of the sparked area. Thus, about 15 pores (excavations) can be counted in the circle with a radius of about 30% of the radius of the sparked area (Figure 14 b). On the right side you can see the characteristic appearance of edging with attack crevices, but with a lower incidence of pitting. The detailed image of the central area in figure 13 reveals the superficial morphology of the crevices, as well as an interconnection between the crevasse in the centre figure 14 and a crevasse located on the left-top.

Also figure 14 reveals the cylindrical morphology of the crevices surrounding the central crevasse. On the other hand, in figure 13, figure 14 and figure 15 results that there is no local area that has not been sparked / sparked. The justification would be that there are no traces of morphological aspects generated by the preparation of the sample, as can be seen at the periphery of the overall images. The image in figure 15 reveals the existence of concentrated attack areas and at the periphery of the sparked area and the lack of a “texture” of attack, of spark.

3.5. Conclusions on microscopic investigation of sparked areas

Comparative investigations of four sparks show the existence of concentrated attack areas, which generate microscopic pits / pores and crevices. The way of generating the pittings seems to be the same, attack on inclusions, which explains their dispositions depending on the sample - crown type at sparks no 2 and no 3 and central agglomeration type at sparks no 1 and no 4. Over a relatively correlated distribution of pittings is superimposed a disordered arrangement of other pittings of comparable dimensions.

Images of the central areas of sparks no 1 and no 2 suggest the existence of an attack texture (figure 4 and figure 7), while figure 11 and especially figure 14 show that there is no morphological texture in the central area. The images of the peripheral areas of the sparked area highlight the scattering of sparks and the existence of cracks on a field attacked in its entirety, but with a lower
intensity. The explanation could be a clear shape of the spark, something in the shape of a shower jet, which has a significant spread of water droplets, which at the incidence with a layer of sand generates a crevice bordered by small excavations. Similarly, the morphological profile generated by a spark can be schematically represented figure 16.

![Figure 16. Schematic representation of the "spark attack" mode](image)

### 3.5.1. Original results of microscopic investigation of sparked areas.
Optical microscopy investigations revealed several aspects that are new for the spectrometric analysis of AISI316L steels, respectively:

1. the sparking area is attacked relatively evenly on 85-90% of the apparent diameter of the sparking spot,
2. at the periphery of the sparked area there is a sudden transition from the uniform attack regime to the non-attacked areas,
3. inclusions and other defects constitute preferential points of attack of sparks and are the cause of the generation of pits and crevices in the sparked area,
4. it has been estimated that a spark (a discharge channel) attacks an area of about 300 μm²,
5. some overview images suggest an nonhomogeneous attack caused by the specific distribution of the spark channels, respectively the profile of the attack intensity is of the form given in figure 17.

![Figure 17. Schematic representation of the dependence of the intensity of the spark attack, in the sparked area, depending on the radius.](image)

### 4. Scanning electron microscopy investigations.
The sample under investigation was the sparked area no.1. The overview of the sparked area obtained in the SE-Secondary Electrons mode is shown in figure 18.

![Figure 18. a). Sparkprint fingerprint overview, b). image with markers.](image)
From figure 18 shows very clearly the overall morphology of the sparkprint, respectively it consists of an approximately circular central area (1, figure 18 b), in which the incidence of sparks (individual) destroyed the pre-existing structure. The area has the appearance of a muddy ground where it rained with stone. This area has a diameter of about 2 mm. Circumscribed to this disc is a crown with a side of about 1 mm (2, figure 18 b), in which the incidence of bombardment with sparks is reduced, but in this crown there are depressions (pits or crevices) whose genesis can most likely be attributed to fixed discharges channels. Why they settled in certain areas remains to be studied/researched.

Elemental Analysis by Energy Dispersive X-ray Fluorescence (ED-XRF) can be correlated with BS and COMPO investigations to estimate the combined composition-segregation effect, or distillation of elements in the spark printed surface. Therefore, it was decided that the investigated SEM areas should be analyzed and ED-XRF. Thus, in figure 19 is presented the emission spectrum of the characteristic X-rays induced by the incident electron beam. From figure 19 it results that in the sparked area are found all the main alloying elements of AISI 316L steel, with intensities proportional to the concentrations of the respective elements.

![ED-XRF spectrum obtained on the whole sparkprint fingerprint](image1)

| Elements | C(%) |
|----------|------|
| Cr       | 17.32|
| Ni       | 10.07|
| Mo       | 1.39 |
| Mn       | 1.26 |
| Si       | 0.62 |
| Cu       | 1.3  |
| O        | 2.34 |
| S        | 0.42 |
| Fe       | 65.28|
| Total    | 100  |

The mass concentration C (%) dosed by the ED-XRF technique corresponding to the spectrum in figure 19 is given in Table 1. The analysis of the values presented in Table 1 will be done later, by correlation with the values obtained on smaller areas, from various areas of the sparking area. Several images were purchased from the central area of the sparked area, of which two images obtained in SE mode and cloned images obtained in BSE mode are rendered. BSE images are intended to reveal some changes in chemical homogeneity in the target area.

![a). SE image from the central area of sparkprint fingerprint](image2)

![b). Cloned BSE image of the image in Figure 20a.](image3)
The image in figure 20 a) reveals the morphological aspects in the central area of the sparking area. The image in figure 20 can be generated by bombarding a pasty surface with heavy solid particles. Consequently, the image clearly shows that the central area reached the state of total superficial melting, and the bombardment captured in the figure was performed on a molten surface but with a fairly high viscosity, i.e. the surface was and was not melted.

In conclusion, the central area has the appearance of a quasi-melted area, located from a thermodynamic point of view, in a fluctuating equilibrium. The spark process practically leads to the formation of a molten central area with a diameter of no more than 4 mm, placed on a massive cold sample, which gives rise to two antagonistic processes: a.) smelting with energy input from sparks and b.) rapid or very rapid cooling, with heat transfer to the sample. Thus, in the sparking process, at least in the centre of the sparked area, a completely melted film is obtained, or at least in a viscous state.

The ED-XRF investigations performed on the central area Table 2 Show that the X-ray spectra are quasi-identical figure 19 and figure 22, and the elemental concentrations differ significantly for the elements Ni, Cu, O and S. The measured concentration differences throughout the sparked area and those measured on the central area, they can be adequately discriminated only if the extended uncertainty U (95%) can be estimated.

| Elements | C(%) |
|----------|------|
| Cr       | 17.58|
| Ni       | 9.43 |
| Mo       | 1.5  |
| Mn       | 1.42 |
| Si       | 0.51 |
| Cu       | 1.66 |
| O        | 1.83 |
| S        | 0.18 |
| Fe       | 65.89|
| Total    | 100  |

**Figure 22.** ED-XRF spectrum obtained on the central area of sparkprint fingerprint

**Table 2.** Related concentrations Figure 22.

The SEM images of the median area (figure 23 and figure 24 ) show morphological aspects that differ significantly from those revealed by the images in the central area of the spot, respectively the middle area has a much rougher appearance due to the impact of fewer sparks, which determined a lower heating of the area and implicitly the impact of the spark on a semi-melted material or even in solid state.

**Figure 23.** SE image of the centre of the sparkprint fingerprint.

**Figure 24.** SE image, in detail, in the middle area of the sparkprint fingerprint.
To explain how to form large craters with "royal crown" type collars (figure 24 bottom, middle position) there are two alternatives: 1. - the craters were formed following a massive hit with a corpuscle that has a diameter of about 10-15 \( \mu \text{m} \). 2. - the craters were formed by repeated attack, followed by the cooling of the melt as it flowed on the walls of the crater and solidified, giving the appearance of uniformity, or glazing of the cavities formed. Another important aspect is the formation of molten metal splashes that solidify in various positions.

What must be kept in mind is that the melting and solidification process is fast. In conclusion, the median area has specific morphological aspects, which differentiate it from the central area, respectively has a much more fibrous appearance, due to lower surface melting compared to the central area. This fact is also supported by the existence of molten metal islands formed in this area (Figure 23).

| Elements | C(%) |
|----------|------|
| Cr       | 17,6 |
| Ni       | 9,4  |
| Mo       | 1,5  |
| Mn       | 1,4  |
| Si       | 0,51 |
| Cu       | 1,7  |
| O        | 1,8  |
| S        | 0,2  |
| Fe       | 65,9 |
| Total    | 100  |

Figure 25. ED-XRF spectrum obtained on the median area of the sparkprint.

From the point of view of affecting the chemical composition, no pertinent differentiations can be made, because the ED-XRF spectrum from figure 25 and the data from Table 3 are almost identical to those revealed by figure 22, respectively Table 2. In figure 26 is presented an edifying image regarding the peripheral area of the sparked area, which reveals in the lower left corner, the morphological aspect of the area not attacked by sparks than in a scattered way. In the mentioned area can be identified some obvious cylindrical pits or craters that reveal that an incident discharge channel on an unmelted area has the power to expel the material but also to melt and create the specific collar (figure 26-lateral left).

The image in figure 26 undoubtedly highlights a clear delimitation between the sparked spot and the non-sparked area. It can be stated that at the periphery of the sparked spot there is a crown with a width of about 200 \( \mu \text{m} \), in which the incidence of sparks is lower than in the adjacent inner area. In the marginal crown is identified a molten area of irregular shape with an apparent diameter of about
100 μm, on the right flank of a ditch resulting from the initial processing of the sample. There is basically a series of such spots irregularly arranged in the peripheral crown.

Inside the sparking area, the right side of the image in figure 26, four molten areas of irregular shape are observed. The central areas of the melts are morphologically homogeneous, which can be explained in two ways: a) the melts formed on smaller areas and merged because they had the time needed for this process; b) an area was melted simultaneously and subsequently, melting craters were created by bombardment (see figure 26).

The first hypothesis seems to be supported by the molten peripheral formations, which have partially adhered to the central melt. Finding the mechanism for the formation of molten areas can be an important step in elucidating the mechanism of the interaction of sparks with the sample. Outside the molten areas of figure 26, individualized craters of small area (Φ 3±5 μm) are observed, but especially larger craters (Φ 10±20 μm), which completely cover the surface inside the circle of delimiting the sparkle imprint by overlapping or juxtaposing.

The image in figure 27 brings new details regarding the spark of the stainless steel samples, respectively:
1. pits with diameters of 1-2 μm arranged in formations at the bottom of the molten central area shall be identified,
2. in the central molten area there is a formation of small craters with a diameter of 1-2 μm,
3. small craters or pits do not form collars if they have diameters less than 3μm,
4. there are no large craters in the molten areas, so they no longer initiate discharges,
5. the areas adjacent to the molten areas are covered with a carpet of craters with diameters of 10-20 μm. This makes plausible the hypothesis that individual discharges are initiated on the collars of the previous craters, which create field concentrators and at the same time are warmer, which makes them more electron-emissive than the other areas. This attracts sparks as lightning is attracted.

5. Conclusions on SEM investigations
The images presented above undoubtedly highlight the explosive nature of individual craters. Also, the respective figures confirm the existence of some channels somewhat quantified according to the size of the surface diameter of the generated pitting, respectively:
- 1±2 μm - craters with insignificant impact, without collar,
- 2.5±3.5 μm - the most representative in terms of shock wave detection and the formation of compact wave collars,
- 8±10 μm - generate extensive surface craters, in already molten areas, or generate crevices at the periphery of the sparked domain.

Spark channels with larger diameters may form, generating large molten areas, but this is unlikely. Most likely, the strongly melted areas are due to the repeated impact of some sparks channels with diameters of about 10 μm. All experimental evidence of optical and electron microscopy leads to the idea of a local discharge. The discharges are made through channels with relatively large diameters, > 2 μm, which remains to be explained. A discharge channel generates shock waves that expel material in the form of excited atoms, ions, but also clusters and particles with diameters < 2 μm. The particles deposit at the periphery of the spark and generate a "peach peel"-shaped surface. "Peach peel" is the reason why the crown of the spark appears as black. In fact, the crown does not contain anything that is black or absorbs light, but the light is dispersed, or rather diffused, randomly scattered, at an incidence on an area with scattering centers of 0,2±5 μm, which break the wave front.

Basically, at the level of the imprint, from the centre to the outside, the density of the diffusive particles increases (which break the incident wave front) and practically the light is no longer reflected. What happens at this level remains to be studied. What is certain is that the black appearance of the sparking crown or the appearance of a smoky stain from the centre to the outside is given by the deposited metal particles. On the other hand, the central area of the sample reflects light, because it has smooth areas, with dimensions of 100-200 μm, which form tiny mirrors that reflect, relatively diffuse,
but reflect light. Thus, through these investigations was found, among other elucidated aspects, the explanation of the black colour of the crown of the sparking area.

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