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Additional Information

# Correlations between acoustic and electrochemical measurements for metallic corrosion on steel strings used in guitars

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## Abstract

The corrosion state of steel guitar strings and their morphology were evaluated according to exposure time to artificial human sweat solution. The instantaneous corrosion rate was evaluated using polarisation resistance. Electrochemical impedance spectroscopy was used to measure the strings' state due to corrosion in artificial human sweat solution. Modification of vibroacustic parameters was analyzed: changes in harmonic content of sound were studied by means of the Fast Fourier Transform and spectrograms. The correlation between corrosion and acoustic measurements was established in the successive stages of corrosion. Thus, the strings' acoustic properties could be modified by means of controlled corrosion processes.

Keywords: steel corrosion, EIS, polarisation, string, acoustic emission

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## 1. Introduction

Electric and acoustic guitar players usually notice how sound qualities of their instruments are becoming modified as the strings are becoming corroded by daily use.

This process is attributable to daily human sweat, which is a well known observable fact that every musician usually experiences, which means that corroded strings have to be replaced with a new set in order to keep the high frequencies and volume at their best sound quality [1].

Research works give us a small number of references working with guitar steel strings and the consequences of human sweat exposure on them. Dissolved metal ions released from guitar strings have been observed in an artificial human sweat solution by scientists of Zagreb's University [2-4]. Incidence of possible allergic reactions caused by nickel metal ions has also been studied. A complete set of six commercial D'Addario electric guitar strings were tested, the whole set was formed by two kinds of strings. Three of them were W type (Wrap), these were: E6, A5 and D4, formed by two steel wires, one of them made of hexagonal cross section and tin covered, and the other wire made of steel with a nickel coated round section. The last wire remains wrapped around the one with hexagonal cross section. The remaining three strings: G3, B2, E1, are single formed with one single steel wire that has circular cross section and is tin coated [5]. In 2009, experiments with E6 and D4 strings were reported by this team of scientists [2]. In 2010, the experiments were carried out with the complete set of six strings [4]. The strings were characterized by means of an optical microscope and scanning electron microscope (SEM). Their chemical composition was determined by inductively coupled plasma-optical emission spectrometry (ICP-OES), and additionally with atomic absorption spectrometry (AAS). Chemical composition of tin and nickel coatings were obtained in both types of strings, wrap and single ones by scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM/EDX). Nickel coating thickness was obtained before and after a controlled corrosion process inside an artificial human sweat solution of 10 ml (EN 1811.1999). Over a period of one month this experiment was carried out four times. At the end of the first week, amounts of metallic ions released in the sweat solution:  $Mn^{2+}$ ,  $Ni^{2+}$ ,  $Si^{4+}$ ,  $Sn^{2+}$  and  $Fe^{3+}$  were obtained by means of ICP-OES. This was done also at the end of the second week and the fourth week.

The starting point is to consider that the strings' chemical composition has an influence on sound, and therefore strings sound qualities will be affected by the corrosion process caused by human sweat [5].

D4 Commercial D`Addario electric guitar strings are chosen for this work, as these were the ones used by the scientists of Zagreb's University [2-4] in their experimental works.

The purpose of this article is to establish a quantitative relationship between damage produced by metallic corrosion in guitar strings and how it causes important changes in their acoustic qualities. This work could be useful to analyze how corrosion processes

lead to changes in the acoustic behaviour, as well as to understand the effect of corrosion in the working life of strings.

The aim of this experimental work has been to simulate real conditions in corrosion processes in guitar strings. The corrosion state of each metallic component of the strings was evaluated depending on exposure time to corrosive agents. A qualitative relationship between corrosion and acoustic measurements was established. The guitar strings' morphology subjected to the corrosion process was also analyzed.

## 2. Material and methods

#### 2.1. Materials and chemicals

Sodium acetate anhydride (purum) was purchased from Probus. Ascorbic acid (99%), 1,10-phenantroline 1-hydrate (100%), ammonia (25%), sodium chloride (99.5%), urea crystal (99-100%), acetone (99.5%) and nitric acid (65%) were from Panreac. Lactic acid (90%) was supplied by Fluka.

Water was obtained from a Millipore-Milli-Q System with a resistivity near to  $18.2 \text{ M}\Omega$  cm. All potentials were referred to the Ag/AgCl (3 M KCl) electrode. Oxygen was removed by bubbling N<sub>2</sub> gas through the solutions.

The guitar strings chosen consisted of two wires. The first, which supports the axial force, is the central core of cold drawn high-carbon music wire. This wire is tin coated and has a hexagonal cross section. The second wire has circular cross section and is made of carbon steel. This wire is nickel plated and it is coiled on the first one [5]. Fig. 1 shows a schematic view of the string [2]. Diameter of the strings is 0.61 mm. Chemical composition of the new string core was in wt. %: Fe 98.77, Mn 0.52, C 0.41, Si 0.30.

Likewise chemical composition of wrap was in wt. %: Fe 73.95, Si 15.78, C 10.56.

#### 2.2. Electrochemical equipment and experimental procedure

The strings were subjected to treatment with acetone in an ultrasonic bath and dried. Then, the strings were treated under controlled corrosion processes, using solutions that simulate the electrolyte composition of human sweat, according to UNE-EN 1811:1999 + A1 and EN ISO 17700. Chemical composition of the artificial human sweat solution was in wt. % in H<sub>2</sub>O: sodium chloride 0.5% (w/w), lactic acid 0.1% (w/w) and urea 0.1% (w/w). Dilute ammonia 1.0% (w/w) was added until a pH 6.50  $\pm$  0.1. In this work, the concentration of the artificial sweat solution was diluted to 1/10. Previous experimental results showed that the strings were broken at 7 days of controlled corrosion process. The new solution gave a longer period of corrosion time to obtain enough experimental information. In each experimental process, three silicone tubes and three equal strings were used. Each silicone tube was hermetically sealed, housing inside the string under analysis and the artificial sweat solution. Fig. 2 shows this experimental setup. A pair of steel clamps was located at the end of the tubes. A length of 44 cm of string was immersed in artificial sweat solution. Each tube individually stored a volume of 5.5 ml of artificial sweat electrolyte.

The strings were taken out of the solutions at different times, specifically at 0, 3, 7, 14 and 17 days. The following measurements were carried out:

- "Dry" measurements to estimate the electronic resistivity of the strings longitudinally. A pair of tweezers connected to an Ecochemie Autolab PGSTAT302 potentiostat/galvanostat was used.
- 2) The instantaneous corrosion rate of the samples immersed in artificial sweat solution was obtained using the polarization resistance technique (Rp). At the end of the experimental period, polarization curves were also plotted.
- 3) Electrical characterization by electrochemical impedance spectroscopy (EIS).
- 4) Gravimetric measurements.
- 5) A Zeiss Ultra 55 FESEM was used to observe the morphology of the samples using an acceleration voltage of 3kV. Energie dispersive X-ray (EDX) measurements were performed between 0 and 20 kV.
- 6) Degree of corrosion in correlation with vibroacoustic properties.

To do the measurements in solution, the extracted guitar strings were immersed in 30 ml of artificial sweat solution. Measurements were carried out in a standard three-electrode configuration cell. A platinum wire as counter electrode and an Ag/AgCl (3M KCl) reference electrode were used. The Rp measurements were carried out at  $1 \text{mVs}^{-1}$  scan rate. An Ecochemie Autolab PGSTAT302 potentiostat/galvanostat was employed to perform EIS measurements in the  $10^4$  to  $10^{-2}$  Hz frequency range. The amplitude of the sinusoidal voltage employed was  $\pm 10 \text{ mV}$ . Each measurement was carried out at a constant potential equal to the stabilized open circuit potential at the beginning of the experiment. To analyze quantitatively the electrochemical behaviour of the samples, the experimental impedance data were modelled and fitted to data generated by using suitable electrical equivalent circuits and a non-linear least squares fitting minimization method included in the ZPlot/ZView software (3.1c version) from Scribner Associates Inc, Southern Pines, NC, USA.

Gravimetric measurements were taken to determine the weight loss of the strings as a result of corrosion, and for this purpose, after each stage of corrosion, the sweat electrolyte was carefully extracted from the silicone tubes and it was allowed to cool until later use. The strings were taken out of the tubes and were dried and their weight was measured. Electrochemical and acoustic measurements were taken finally.

#### 2.3. Acoustic equipment and experimental procedure

After the electrochemical measurements, acoustic pressure were taken to characterize the vibration behaviour of the strings. Measurements were recorded in a semi anechoic chamber.

A prototype guitar fingerboard was designed specifically for the requirements of this experiment (Fig. 3).

The string is pinned in both ends and has a length of 647.7 mm. A Load Cell sensor was placed to measure the string force variation. The string is tensioned until 89 N to achieve the first mode of vibration at 147Hz. The string is excited in a location 92.5 mm at the one both, where there are not nodal points of the 6<sup>th</sup> first's vibration modes.

Sound pressure measurements were taken in a semi anechoic environment, using a G.R.A.S. 40 AF omnidirectional open field pressure microphone. The signal was amplified and recorded by a National instruments USB 9234 sound card. For signal acquisition and analysis software Matlab functions were used. Sound pressure measurements were processed to determine their frequency content by FFT (Fast Fourier Transform), thus changes in the harmonic content of sound strings were studied with the successive stages of corrosion. Since FFT does not provide information on the time axis, the spectrogram was also obtained and it showed how each frequency varies versus time.

#### 3. Results and discussion

#### 3.1. Electrochemical analysis

Fig. 4 shows the electrical resistivity graph of "dry" measurements obtained after the experimental process for 0, 3, 7, 14 and 17 days inside the artificial sweat solution. The experimental procedure was carried out with three strings of identical characteristics. It is important to note that electrical resistivity increased due to the development of oxides, and due to the reduction of the real area of the string. This can be observed in section 3.3 (Fig. 13), which shows FESEM microphotographs.

Fig. 5 shows Rp measurements of the strings in the synthetic sweat solution. It is noticeable that the corrosive attack starts gradually and becomes more intense until it levels out at a stable value.

When the complete series of Rp measurements with the strings were carried out, Tafel plots [6] were obtained for the guitar strings in synthetic sweat solution. In order to carry out the Tafel tests, firstly the string was polarized  $\pm 250$ mV from the open circuit potential. The scan rate was 1mVs<sup>-1</sup>. Taking into account that the geometric area of the strings was 0.622 cm<sup>2</sup>, the data obtained from Tafel plots (figure not shown) were as follows:  $i_{corr} = 42.9 \ \mu A/cm^2$ , cathodic Tafel slope,  $b_c = 156 \ mV/dec$ , anodic Tafel slope,  $b_a = 403 \ mV/dec$ ,  $E_{corr} = -395 \ mV$ . B<sub>c</sub> and  $b_a$  values were applied in the equation: B =  $(b_c \ x \ b_a)/(2.3(b_c+b_a))$ , which gave the value of B = 48.9 mV. The data obtained were

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consistent since they fall within the range of appropriate values for steels with nickel materials [7]. In addition, it is necessary to highlight that the samples showed a tendency toward passivity due to a higher value of  $b_a$  in comparison with  $b_c$  [8]. Finally, the  $R_p$  value was obtained from B:  $R_p = B / i_{corr} = 1140$  ohms cm<sup>2</sup>. The  $R_p$  value is consistent with those data (Fig. 5) obtained using the technique of polarization resistance  $R_p$ .

In order to characterize the electrical behaviour of the strings ("dry" measurements) by EIS, the equivalent circuit shown in Fig. 6 was proposed. As it was mentioned previously, the structure of the string consists of two steel wires, one of them made with a hexagonal cross section and tin coated, and the other wire made of steel and nickel coated covers the hexagonal core. So it is possible to identify the mentioned structure of the guitar string with the equivalent circuit shown in Fig. 6. In this figure,  $R_a$  corresponds to the steel core resistance,  $R_{ind}$  to the resistance associated with the inductance and L to the inductance, both  $R_{ind}$  and L referred to the wrapped wire.

Fig. 7 and Fig. 8 show the Nyquist and Bode plots for the longitudinal analysis ("dry" measurements) before immersing the string within the synthetic sweat solution.

A clear inductive behaviour can be seen from the experimental data. This was as expected because of the geometric characteristics of the strings. The Nyquist diagram shows that the inductive reactance decreases as the frequency reaches lower values. Therefore, the data in the phase versus frequency plot in Fig. 8 gradually acquires a more resistive behaviour at lower frequencies (0° for the phase). Bode plots show that the impedance modulus was taking smaller values as frequency decreases. Below 1000 Hz the impedance module stabilized with a pure resistive value at  $6.2 \cdot 10^{-5}$  ohms.

Table 1 shows the values obtained for the longitudinal analysis ("dry" measurements) by fitting experimental data to the electrical circuit shown in Fig. 6.

These values refer to  $1 \text{ cm}^2$  of cross section and 1 cm of string length. Every measurement was taken after the corrosive attack, for 3, 7, 14 and 17 days respectively. Initial data refer to the guitar string without synthetic sweat solution treatment. As it can be seen in Fig. 9, the resistance ( $R_{ind}$ ) associated with the inductance of the wrapped wire clearly increases. This is due to the oxidation which the coil is undergoing. Similarly, the  $R_a$  values also tend to increase, although less than the coil resistance, because the steel core does not suffer such a severe corrosive attack. As it can be seen in Table 1, the inductance variations obtained showed a slight increase, probably due to changes in the geometry and chemical composition of the steel wire.

In order to simulate human sweat conditions, the strings were immersed in synthetic sweat solutions. EIS measurements were made at the end of the 3, 7, 14 and 17 day. The presence of numerous micro-pores, irregularities and anomalies were found in the morphology of the electrode, as it can be seen in section 3.3. Fig. 10 shows the Nyquist plots for the string immersed in synthetic sweat solution. The characteristic semicircles are a little flattened. The results have led to use an equivalent circuit which fits all the irregularities provided in the experimental conditions. Constant phase elements (CPEs)

have been introduced to simulate the capacitive behaviour of the capacitors and the diffusion of electro-active ions. These elements are used when the behaviour of the studied material is not ideal, showing anomalies, and irregular porosity. The impedance of the CPE is defined as  $Z_{CPE} = A^{-1}(j\omega)^{-\alpha}$ , where  $\alpha$  is a fractional exponent, having values between 0 and 1;  $j = \sqrt{(-1)}$ ;  $\omega = 2\pi f$  is the angular frequency and A is a frequency independent parameter. For  $\alpha = 0$ , the CPE describes an ideal resistor; for  $\alpha = 1$ , it describes an ideal capacitor (A=C); for  $\alpha = 0.5$ , it represents homogeneous semi-infinite diffusion [9, 10].

For the string in synthetic sweat solution the following contributions can be expected:

- Re: Electrolyte resistance.

- Rg: Electronic resistance of the D4 guitar string. CPEg: non-ideal capacitance in parallel associated with Rg to account for dielectric properties of the guitar string. This non-ideal capacitance could be negligible. Physically, metallic guitar string would act as a resistance more than a dielectric material. Rg values (<<1, resistivity  $6.2 \cdot 10^{-5} \Omega \cdot cm$ ) are negligible in comparison with Re, since the string is a metallic material.

- L: Inductance due to the wrapped wire of the guitar string.

- Rtc: Ion–electron charge transfer resistance at the guitar string/sweat solution interface with redox species. CPEdc: non-ideal capacitance corresponding to the guitar string/sweat solution interface.

- Rd: Diffusion resistance for semi-infinite linear diffusion. CPEd-T: non-ideal capacitance corresponding to semi-infinite linear diffusion. CPEd-P: CPE coefficient near to 0.5 for semi-infinite linear diffusion [9, 10].

Inductive behaviour could not be seen in the Nyquist plot of Fig. 10. Therefore, this element was not taken into account for the equivalent circuit. The equivalent circuit proposed to the corrosion process is shown in Fig. 11.

Table 2 shows the fitting carried out using the proposed equivalent circuit. The values of CPEd-P were close to 0.5. Therefore, these data corroborate that a diffusion process is being measured. Rtc and Rd provided the upper values of the whole electric response. Between the two values, the greater one was Rd (diffusion resistance). Therefore, diffusion was the rate-limiting step in this corrosion process. The Rtc (ion–electron charge transfer resistance at the guitar string/sweat solution interface) tends to diminish. Corrosion attack is more severe and Rtc was minor.  $CPE_{tc}$ -P also tends to diminish. This fact means that more micro-pores, irregularities and anomalies were found in the structure morphology of the electrode due to corrosion process. Rd (diffusion resistance) tends to diminish as a consequence of more severe corrosive attack on the strings.

#### 3.2. Measurement of weight loss

Fig. 12 shows the weight measurements of the strings. These values were obtained at the end of each experimental process to quantify the weight loss caused by corrosion. It can be appreciated that the string suffered a slight mass loss resulting from the decomposition of the metal. Consequently, the string diameter decreased due to the corrosion process.

This was in concordance with Fig. 9 and Table 1 where the resistance (Rind) associated with the inductance of the wrapped wire increased. The Ra (guitar string steel core) values also tend to increase, a consequence of a minor cross section diameter of the guitar strings due to corrosion process.

The data obtained, by energy dispersive X-ray (EDX) analysis on the seventeenth day of the corrosion process, shows a huge difference in wt. % of oxygen concentration between the values obtained for the core (31.33%) and the winding (6.78%). These results imply that corrosion in the core is much more intense than in the winding. Moreover, that significant amount of oxygen obtained shows the formation of iron oxides that remain part of the surface of the string and justifying that there is no significant loss of mass.

3.3. Surface morphology analysis by field emission scanning electronic microscopy (FESEM).

In Fig. 13 (a,c,e) left images show microphotographs which were taken with an original D4 string, likewise (b,d,f) right images show microphotographs which were taken after seventeen days at the end of the experiment using FESEM.

Figures 13a and 13b show a longitudinal fragment of string, while Figures 13c and 13d show a detailed image of the string. Differences between the original state of the new string and its appearance on the 17th day of corrosive attack are remarkable.

Figures 13b and 13d show the characteristic oxide deposit located between the helices of the coil of the guitar string after seventeen days in corrosive solution.

Corrosion likewise produced on the surface of the coil is much smaller than that occurred in the core as can be seen by comparing Figure 13f with Figure 13d.

Figure 13e shows the core of the string in its original state.

Right FESEM microphotographs show a significant amount of oxides leaving from the core string. Inside the guitar string (core rolled by the winding), the oxygen concentration could be lower. Differential aeration corrosion could be present in this

system. Tin and nickel are more noble metals than steel in this medium. A galvanic or two-metal corrosion could also be present in this system. Tin and nickel could especially accelerate corrosion of the core string as EDX analysis showed (wt. % :31.33 oxygen content)

#### 3.4. Acoustic analysis

The string was subjected to a controlled corrosion process in five stages: 0, 3, 7, 14, 17 days. For each stage electrochemical data are measured, but for acoustics data only 0, 3 and 7 days are measured because the string broke due to the corrosion process

Fig. 15 describes the spectrum analysis and the spectrogram for the three stages of the corrosion process.

The Fig. 15a shows the frequency-amplitude response of the string before the application of artificial human sweat treatment. The frequencies of the modes of vibration obtained experimentally are coincident to the expected theoretical values for the vibration string according to the equation (1):

f is the vibration frequency, L is the length of the string, n number mode and is an integer,  $\mu$  is the linear density, and T is the tension of the string.

The Fig. 15d shows the spectrogram, that is the frequency-time response of the string, before the application of artificial human sweat treatment. The first mode of vibration at 147 Hz is clearly the longest in time.

The Figs. 15b and 15c show the frequency-amplitude response of the string in the second stage and third stage. The modes of vibration trend to disappear over 1500 Hz. As for the spectrogram, represented in Figs. 15e and 15f, the duration of the fundamental frequency of 147 Hz shortens in time with the progressing of the corrosion process.

Finally, a correlation between Rp and the amplitude of fundamental frequency (147 Hz) is shown in Fig. 16. The Rp decreased during seven days of test. The amplitude of the fundamental frequency (147 Hz) also similarly decreased for the seven days.

### 4. Conclusions

The results show a progressive loss of the upper vibrational modes of the string and a decreasing duration time of the signal with the progress in the process of controlled corrosion.

Electrical resistivity and EIS measurements ("dry" measurements) on the strings did not show a notable increase of electrical resistivity after the three days of corrosion attack. Measurements of weight loss did not also show a significant weight decrease of the strings due to the corrosion process throughout the first three days under corrosion. In contrast, a drastic decrease (one order of magnitude) was observed for R<sub>p</sub> measurements in synthetic human sweat solution. R<sub>p</sub> measurements in synthetic human sweat provided a suitable method to control the sound characteristics of strings during corrosion processes. An appropriate correlation was obtained between polarization resistance Rp and sound amplitude (FFT) measurements.

In conclusion, electrochemical methods could be applied to evaluate changes in acoustic behaviour of strings of musical instruments by means of a controlled corrosion process.

In fact, at this time, our research team is using the working method detailed in this manuscript to evaluate the effectiveness of various methods to prevent corrosion on the strings of musical instruments.

Two hypotheses were established: Impressed current protection and sacrificial anode protection could diminish the corrosion in guitar strings immersed in artificial human sweat. Preliminary results showed a lower level of corrosion using impressed current cathodic protection.



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## **TABLE CAPTIONS**

Table 1. Values obtained for the EIS equivalent circuit parameters of Fig. 6 during the experimental for D'Addario D4 string obtained in longitudinal analysis and dry

Table 2. Results of the fitting of impedance data corresponding to the equivalent circuit proposed for the corrosion process of Fig. 11.

#### FIGURE CAPTIONS

Fig. 1. FESEM microphotograph of D4 electric guitar string [2].

Fig. 2. D4 guitar string inside silicon tube with artificial sweat solution.

Fig. 3. Guitar fingerboard prototype into a semi anechoic camera.

Fig. 4. Electrical Resistivity graph vs time and data table for D4 string obtained in longitudinal analysis and dry conditions. String length: 70 cm, cross section: 0,003 cm<sup>2</sup>.

Fig. 5. Polarization resistance  $R_p$  vs time graph for D4 string within the synthetic sweat solution.

Fig. 6. EIS analysis equivalent circuit for D4 string obtained in longitudinal analysis and dry conditions.

Fig. 7. Nyquist plot for an original D4 string obtained in longitudinal analysis and dry conditions. The plot shows initial values before immersing the string within the synthetic sweat dissolution. Cross section of the string: 1cm<sup>2</sup>, length: 1 cm.

Fig. 8. Bode plots for an original D4 string obtained in longitudinal analysis and dry conditions. The plot shows initial values before immersing the string within the synthetic sweat dissolution. Cross section of the string: 1cm<sup>2</sup>, length: 1 cm.

Fig. 9.  $R_a$  and  $R_{ind}$  graph for an original D4 string obtained in longitudinal analysis and dry conditions by EIS analysis. Cross section of the string:  $1 \text{ cm}^2$ , length: 1 cm.

Fig. 10. Nyquist plot with zoom image for the analysis of EIS for D4 string obtained within the synthetic sweat dissolution along 3, 7, 14 and 17 days of corrosion process. Cross section of the string: 1cm<sup>2</sup>, length: 1cm.

Fig. 11. EIS analysis equivalent circuit for D4 string obtained within the synthetic sweat dissolution.

Fig. 12. Weigh measurement graph for D4 strings after corrosion process in synthetic sweat solution.

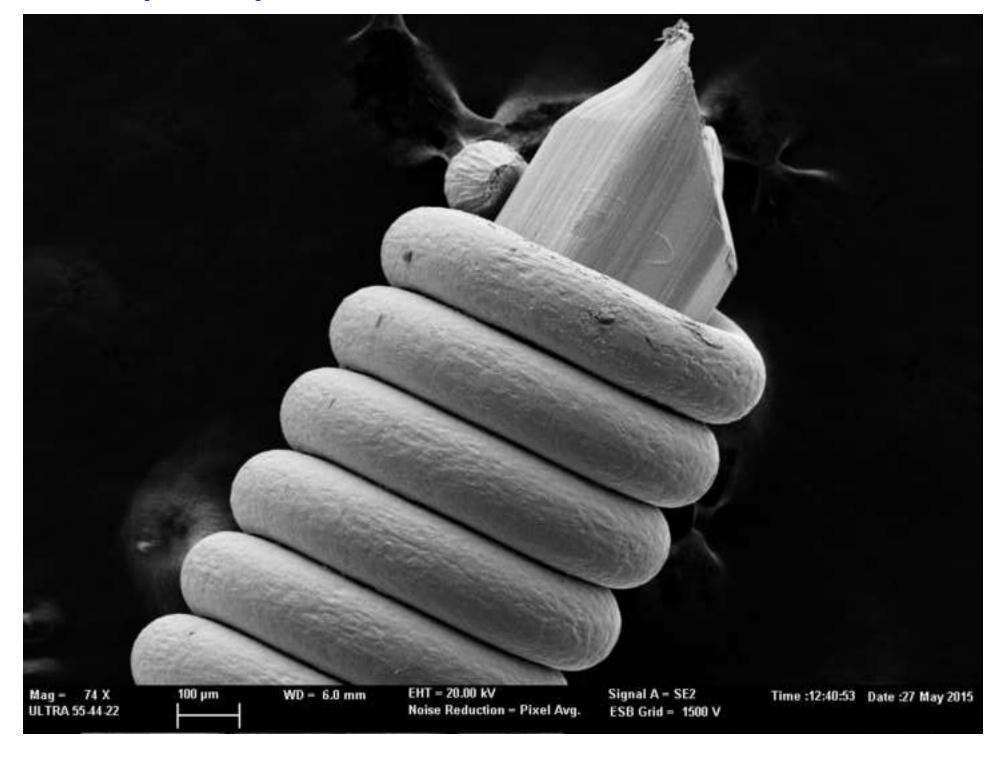
Fig. 13. FESEM microphotographs of a D4 string obtained along the corrosion process in synthetic sweat solution: (a,c,e,g) with an original D4 string, (b,d,f,h) after seventeen days.

Fig. 14 shows the amplitude-time recording of the string before corrosion. As the corrosion process was progressing, a higher decay in the time-amplitude signal was observed.

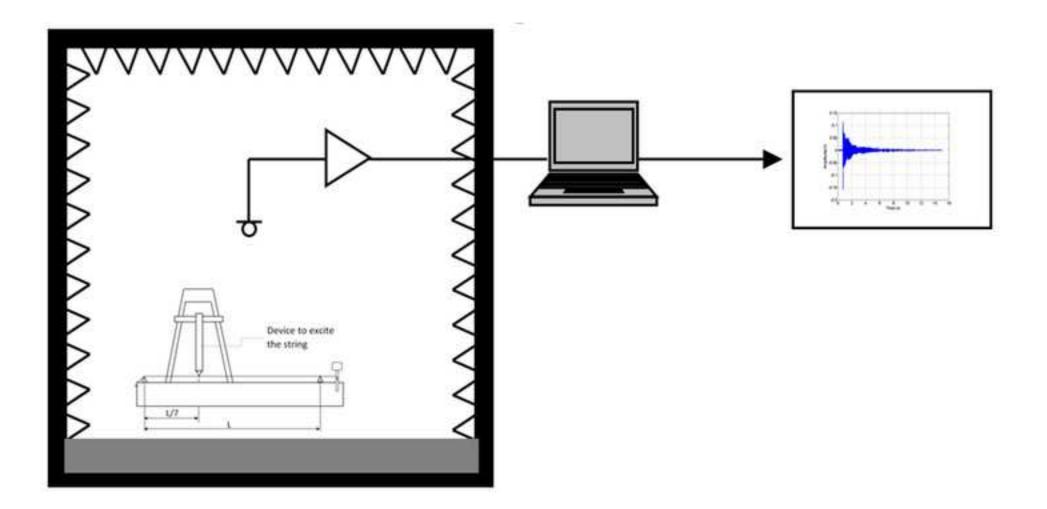
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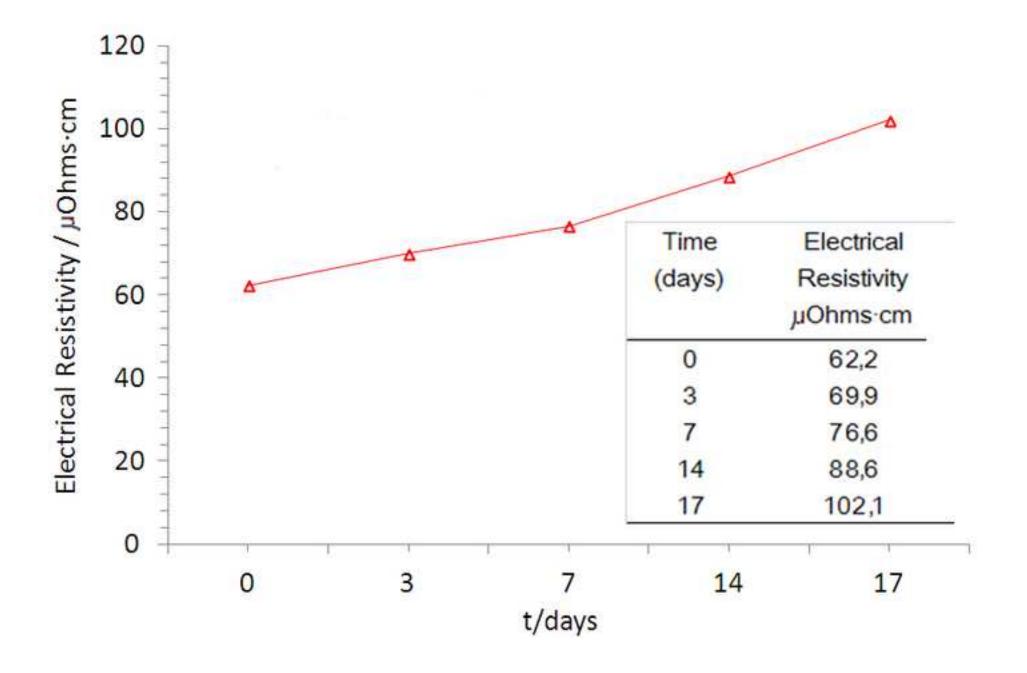
Fig. 15. Spectrum and spectrogram analysis for a D`Addario D4 string along the corrosion process in synthetic sweat solution: (a) Spectrum with an original D4 string, (b) Spectrum after three days, (c) Spectrum after seven days. (d) Spectrogram with an original D4 string, (e) Spectrogram after three days, (f) Spectrogram after seven days.

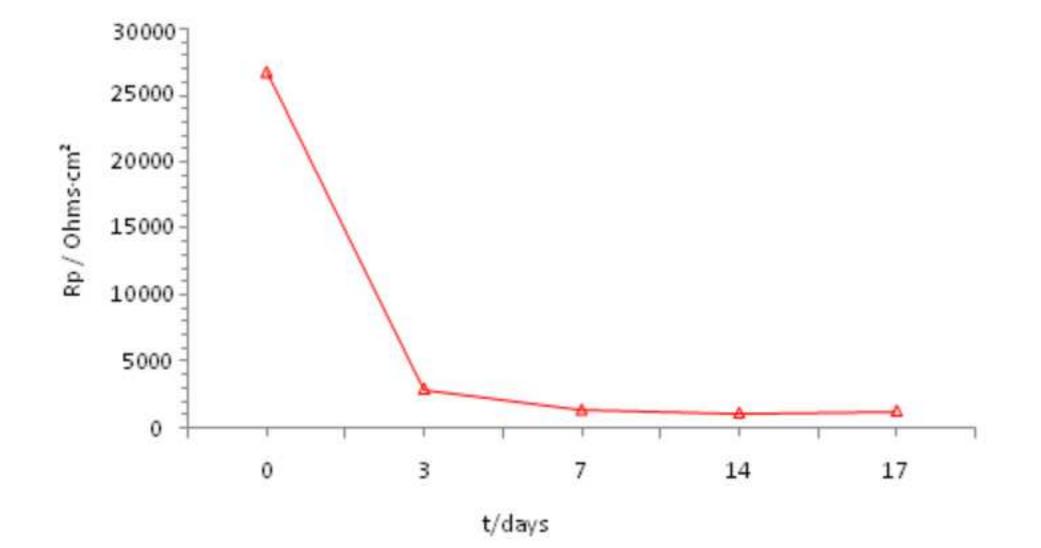
Fig.16 • Polarization resistance Rp vs time for D4 string within the synthetic sweat solution; • Amplitude (FFT) of the fundamental frequency (147 Hz) vs time for D4 string along the corrosion process in synthetic sweat solution.

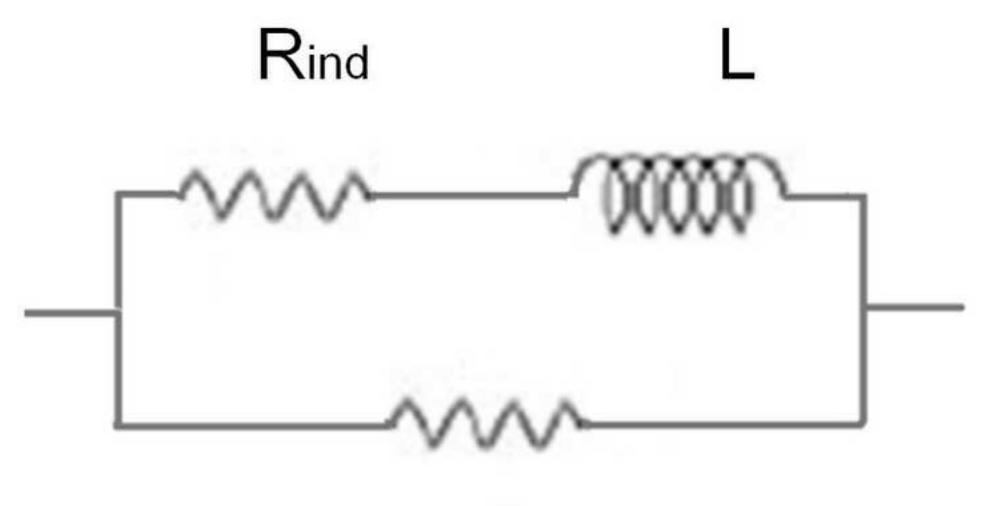




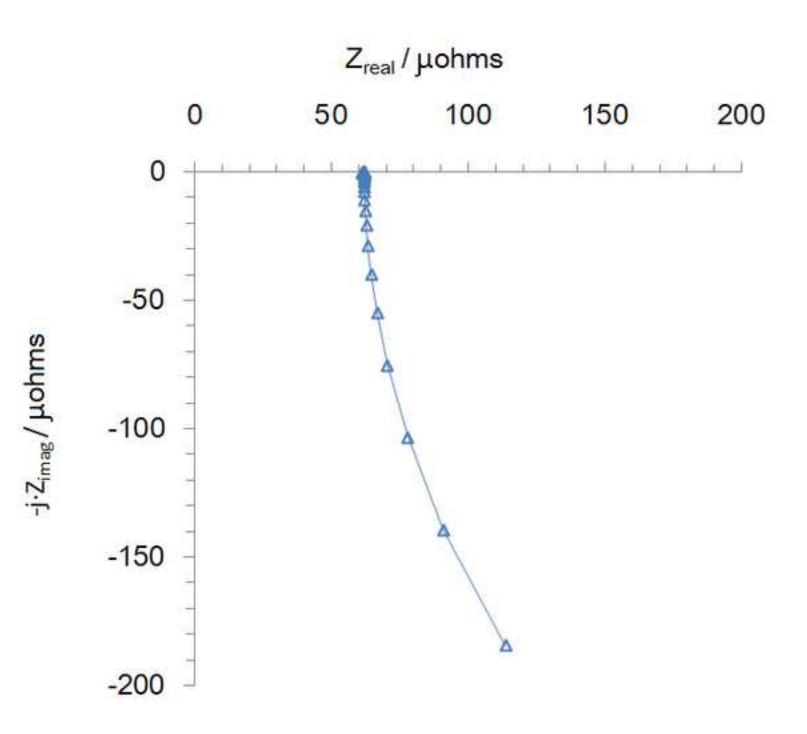


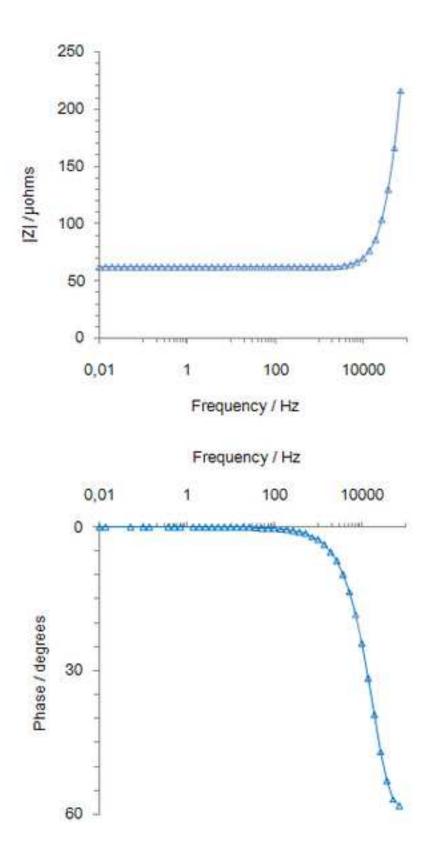


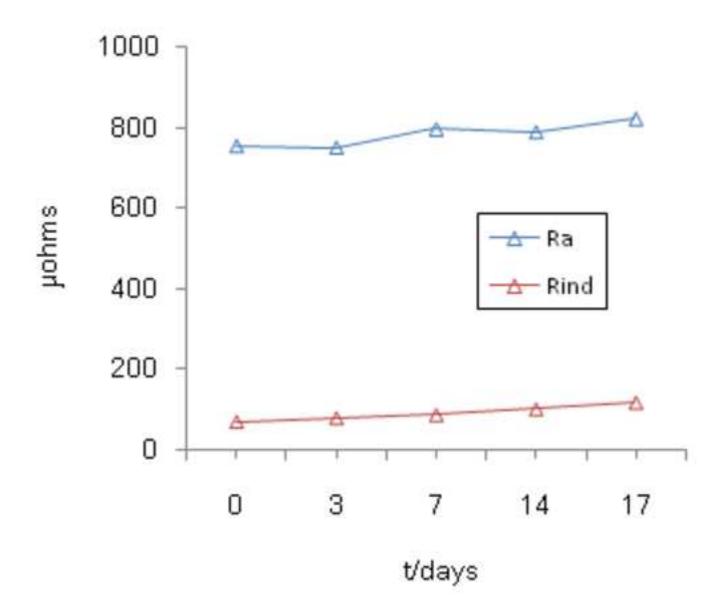


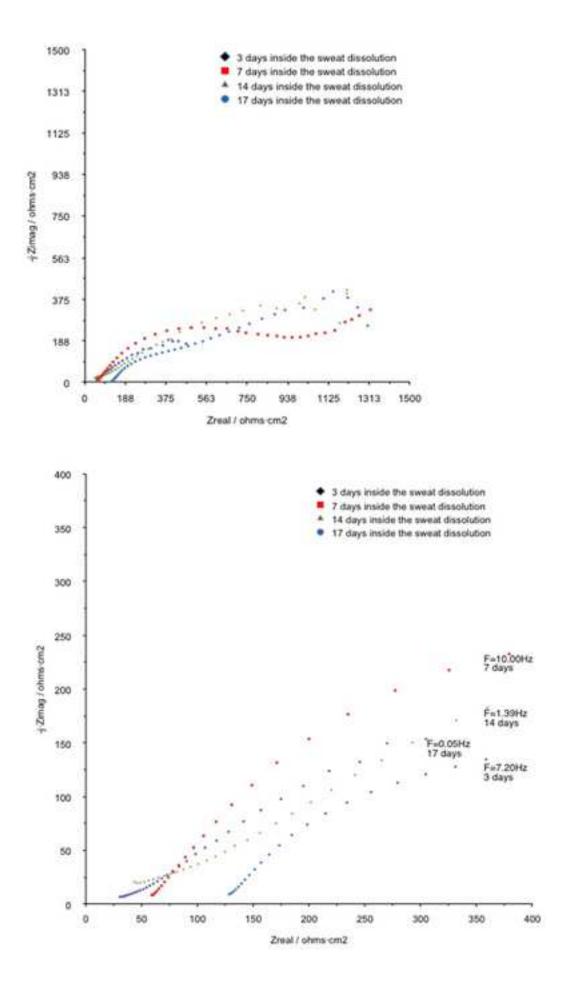


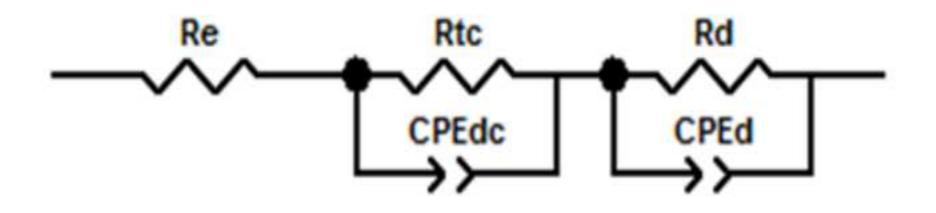
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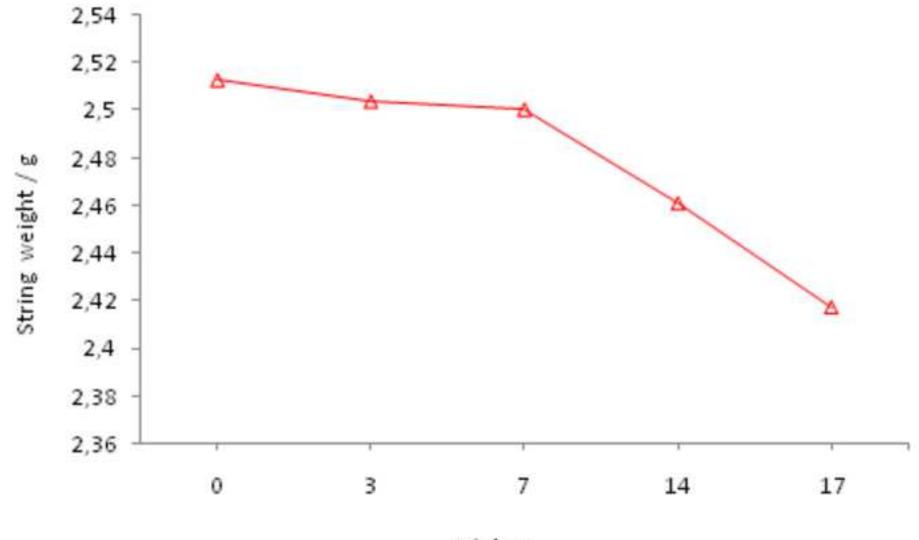




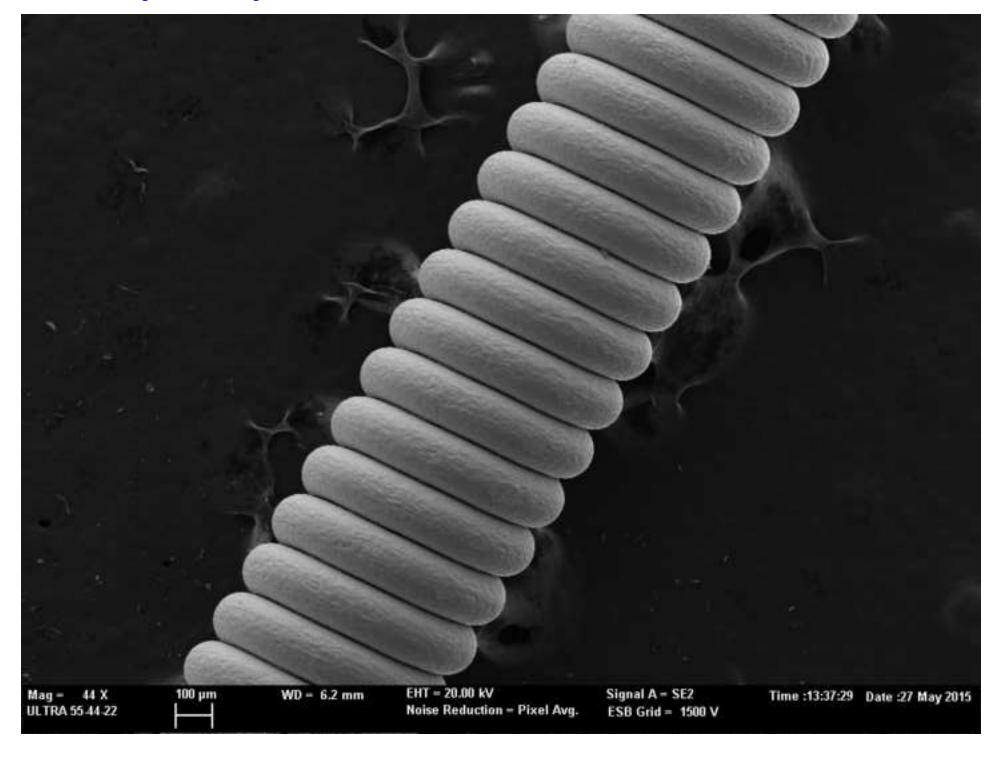


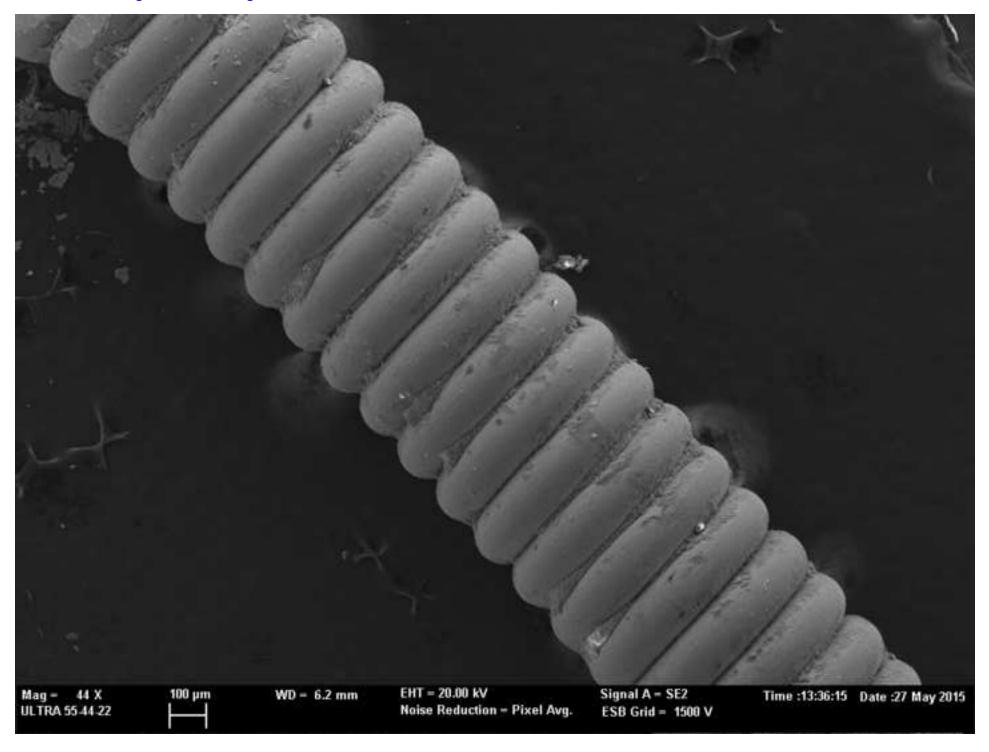


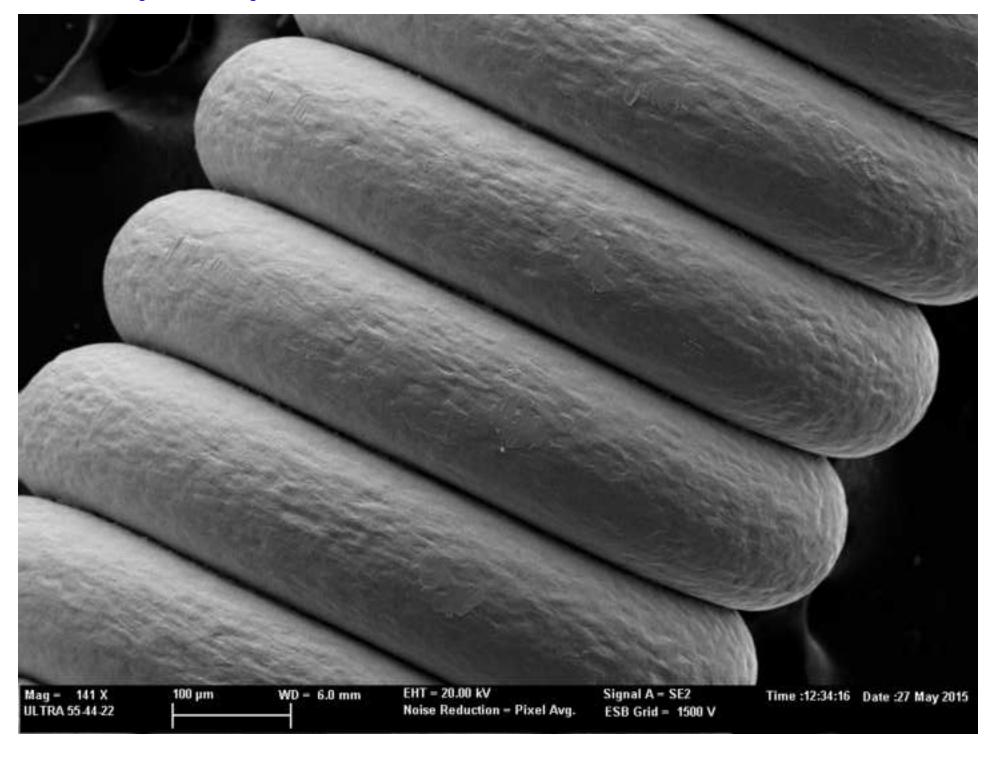


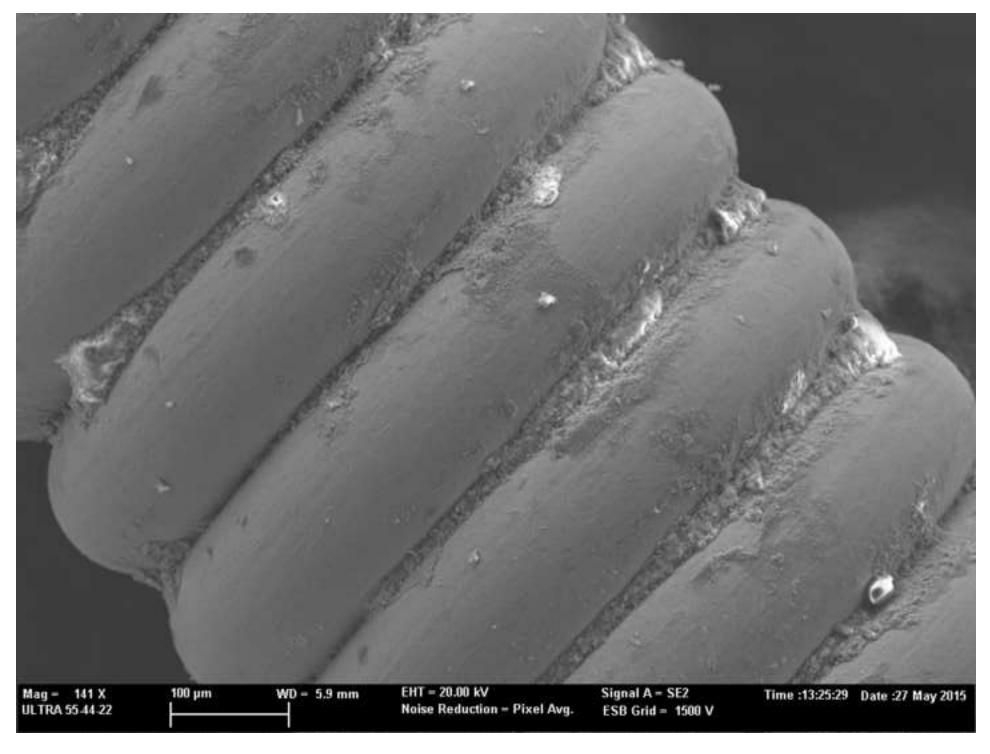


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