Report Prepared by:
Francisco J. Presuel-Moreno
With
Carlos Castaneda
Ingrid Santillan
Dr. Amirkhosro Kazemi
Dr. Fujian Tang

Final Report

Corrosion Prevention of Bridge Tendons Using Flexible Filler Materials
BDV27-977-10

Submitted to
Florida Department of Transportation Research Center
605 Suwannee Street
Tallahassee, Florida 32399

Submitted by
Francisco Presuel-Moreno
Principal Investigator
Department of Ocean and Mechanical Engineering
Center for Marine Materials
Florida Atlantic University - Sea Tech

101 North Beach Road
Dania Beach, Florida 33004

November 2018

Disclaimer

The opinions, findings, and conclusions expressed in this publication are those of the author and not necessarily those of the State of Florida Department of Transportation

Units Conversion Page

SI* (MODERN METRIC) CONVERSION FACTORS

APPROXIMATE CONVERSIONS TO SI UNITS

Symbol	When You Know	Multiply By	To Find	Symbol
		LENGTH		
in	inches	25.4	millimeters	mm
ft	feet	0.305	meters	m
yd	yards	0.914	meters	m
mi	miles	1.61	kilometers	km
		AREA		
in^2	square inches	645.2	square millimeters	mm^2
ft^2	square feet	0.093	square meters	m^2
yd^2	square yard	0.836	square meters	m^2
ac	acres	0.405	hectares	ha
mi^2	square miles	2.59	square kilometers	km^2
		VOLUME		
fl oz	fluid ounces	29.57	milliliters	mL
gal	gallons	3.785	liters	L
ft^3	cubic feet	0.028	cubic meters	m^3
yd^3	cubic yards	0.765	cubic meters	m^3
		NOTE: volumes greater than 1000 L shall be shown in m^3		
		MASS		
oz	ounces	28.35	grams	g
lb	pounds	0.454	kilograms	kg
T	short tons (2000 lb)	0.907	megagrams (or "metric ton")	Mg (or "t")
		TEMPERATURE (exact degrees)		
°F	Fahrenheit	5 (F-32)/9 or (F-32)/1.8	Celsius	°C
		ILLUMINATION		
fc	foot-candles	10.76	lux	lx
fl	foot-Lamberts	3.426	candela/m^2	cd/m^2
		FORCE and PRESSURE or STRESS		
lbf	poundforce	4.45	newtons	N
lbf/in^2	poundforce per square inch	6.89	kilopascals	kPa

APPROXIMATE CONVERSIONS FROM SI UNITS

Symbol	When You Know	Multiply By	To Find	Symbol
		LENGTH		
mm	millimeters	0.039	inches	in
m	meters	3.28	feet	ft
m	meters	1.09	yards	yd
km	kilometers	0.621	miles	mi
		AREA		
mm^2	square millimeters	0.0016	square inches	in^2
m^2	square meters	10.764	square feet	ft^2
m^2	square meters	1.195	square yards	yd^2
ha	hectares	2.47	acres	ac
km^2	square kilometers	0.386	square miles	mi^2
		VOLUME		
mL	milliliters	0.034	fluid ounces	fl oz
L	liters	0.264	gallons	gal
m^3	cubic meters	35.314	cubic feet	ft^3
m^3	cubic meters	1.307	cubic yards	yd^3
		MASS		
g	grams	0.035	ounces	oz
kg	kilograms	2.202	pounds	lb
Mg (or "t")	megagrams (or "metric ton")	1.103	short tons (2000 lb)	T
		TEMPERATURE (exact degrees)		
°C	Celsius	1.8C+32	Fahrenheit	°F
		ILLUMINATION		
lx	lux	0.0929	foot-candles	fc
cd/m^2	candela/m^2	0.2919	foot-Lamberts	fl
		FORCE and PRESSURE or STRESS		
N	newtons	0.225	poundforce	lbf
kPa	kilopascals	0.145	poundforce per square inch	lbf/in^2

*SI is the symbol for the International System of Units. Appropriate rounding should be made to comply with Section 4 of ASTM E380.
(Revised March 2003)

Technical Report Documentation Page

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle Corrosion Prevention of Bridge Tendons Using Flexible Filler Materials		5. Report Date November 2018 6. Performing Organization Code FAU-OE-CMM-08-3
7. Author(s) Francisco J. Presuel-Moreno, Carlos Castaneda, Ingrid Santillan, Dr. Amirkhosro Kazemi, Dr. Fujian Tang.		8. Performing Organization Report No. BDV27-977-10
9. Performing Organization Name and Address Center for Marine Materials Florida Atlantic University – Sea Tech 101 North Beach Road Dania Beach, Florida 33004		10. Work Unit No. (TRAIS) 11. Contract or Grant No. BDV27-977-10
12. Sponsoring Agency Name and Address Florida Department of Transportation 605 Suwannee Street, MS 30 Tallahassee, FL 32399		13. Type of Report and Period Covered Final Report December 21 2015 – November 30, 2018 14. Sponsoring Agency Code
15. Supplementary Notes		

16. Abstract

Over the last few decades, flexible fillers such as greases or waxes have been used in Europe as an alternative to cementitious grout. The Florida Department of Transportation (FDOT) is considering flexible fillers as an alternative filler material for post-tensioned (PT) tendons. In this study, the corrosion resistance of these fillers to protect PT tendons was investigated experimentally. Wire or steel strands were coated with flexible filler materials. Selected samples were contaminated with fungi. Three types of fungi were considered, including *Fusarium oxysporum* (FO), *Penicillium chrysogenum* (PC), and *Aspergillus flavus* (AF). The flexible fillers investigated are commercially available microcrystalline waxes. Five different filler types were investigated. Both exposure and electrochemical corrosion tests were designed and performed. Two exposure conditions were considered, including the direct outdoor exposure test and the indoor exposure test. Electrochemical corrosion tests showed that most of the coated strands had impedances higher than 1 Gohm and galvanic corrosion currents lower than 1 nA. However, for the steel strand coated with filler type 3, an increase in the galvanic corrosion current and a decrease in the impedance were observed after two weeks of tests, indicating corrosion initiation. Direct outdoor exposure tests showed that rust was present on steel single wires coated with type 1, type 2, type 3, type 4, and type 5 filler after 20 days, 43 days, 49 days, 114 days, and 126 days of exposure, respectively. Different from the general corrosion observed on wires subjected to direct outdoor exposure tests, localized corrosion was present on indoor wires which were coated with flexible filler and contaminated with fungi. Larger samples were prepared in which the filler was injected into high density polyethylene or polycarbonate tubes. The samples contained from 3 to 19 steel strands. Most samples were also exposed outdoors, and selected samples were periodically sprayed with solutions containing water, or water-fungi-mix. Solution was sprayed at 3- to 5-weeks intervals. The corrosion extent on these samples was significantly less.

17. Key Word	18. Distribution Statement
Flexible Filler, Microcrystalline Wax, Fungi, Post-tensioned Tendons, Steel Wire.	

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price
Unclassified	Unclassified	310	

Form DOT F 1700.7 (8-72) Reproduction of completed page authorized

Acknowledgments

The authors are indebted to the FDOT State Materials Office (SMO) for assisting in coordinating sample preparation, in particular, Mr. Ivan Lasa and Mr. Ronald Simmons. The authors also acknowledge the assistance from the Marcus H. Ansley Structures Research Center (SRC) for providing flexible fillers and PT steel strands and assisting with flexible filler injection into samples, in particular, William Potter, Bruno Vasconcelos, and several SRC Technicians. The assistance of several students who worked as graduate/undergraduate research assistants at FAU-Sea Tech marine materials and corrosion lab is also acknowledged.

Executive Summary

Flexible filler is a grease or microcrystalline wax derived from petroleum. It was introduced to the U.S. during the early 1950s for mono-strand tendon systems [3]. Compared with cement grout, the main advantages of flexible fillers are shorter construction time, reduced structural depth, increased stiffness, and savings in overall cost. They has been used in enclosed buildings, parking structures, and pressure vessels in the nuclear power industry [4]. In this study, five flexible fillers were investigated, and all were microcrystalline wax materials. There is a need to better understand how flexible fillers would perform in the Florida environment. These flexible filler products are intended to be used as fillers in post-tensioning (PT) tendons to cover the steel strands and prevent corrosion from taking place.

However, corrosion of steel coated with flexible fillers has also been observed. The reasons include water presence (either due to holes/voids and/or improper sealing), poor quality of the flexible filler application, and microorganisms (microbiologically influenced corrosion due to bacteria/fungi) [5]. In cases where strand corrosion was apparent, the attack was generally most advanced within and immediately behind the anchors and progressively moderated relative to the distance inboard. The wire corrosion in the wedge zone can cause disengagement, slippage, load release, and tendon failure [6].

The typical corrosion type in PT tendons coated with flexible filler is microbiologically influenced corrosion (MIC) due to bacterial or fungal contamination. MIC is caused by the presence and activities of microorganisms. MIC is a microbiologically mediated reaction with metal oxides, protective material layers, or formation and dissolution of minerals [7-9]. In this type of corrosion, different bacteria or fungi can develop that later produce an acidic environment that allows corrosion to initiate. The high humidity in the Florida environment might allow fungi to develop and contaminate the filler if not properly covered while awaiting installation or if the anchorage is not properly sealed. To limit the scope of the research, fungi were chosen over bacteria for this investigation. Three types of fungi were considered, including *Fusarium oxysporum* (FO), *Penicillium chrysogenum* (PC), and *Aspergillus flavus* (AF)

Two types of samples were used in this study based on dimension and filler application. Large injected samples were samples with PT steel strands and the duct of at least 2 inches in diameter and at least 2 feet long. The smaller samples were typically filler-coated steel wires (except for injected samples on 0.75-inch diameter polycarbonate with various wires). In some cases, a single coated steel strand (4 feet long) was used as the specimen.

The purpose of this study was to investigate the corrosion performance of steel strands coated with five different flexible fillers as they are exposed to different environments and contaminated with three fungi species. Both steel strands and single steel wire are tested. The methods included contamination tests, exposure tests, and electrochemical corrosion tests.

The worst corrosion was observed on samples exposed outdoors (both coated strands and coated wires) at a partially sheltered location where ocean spray particulates reached the samples on windy days. Moisture from rain and/or storms wetted the samples on occasion.

Forensic analysis, including stereo micrographs and scanning electron microscope, provided evidence of the corrosion extent on steel wires after cleaning. One type of sample was

coated strands sprayed with fungi mix (stored on a PVC pipe segment) along the 4-foot strand length. Spraying took place every five weeks. Other cleaned wires were removed from injected five-inch-long samples (injected with the different fillers in 0.75-inch diameter polycarbonate tubes) after spraying wetting with various solutions.

Corrosion was observed on samples that were exposed both outdoors and indoors in a high humidity environment. The coated strands exposed outdoors and coated wires exposed outdoors experienced the most corrosion and appeared to be more uniformly corroded after cleaning.

Wires exposed indoors to high humidity also experienced corrosion but to a lesser extent.

Larger samples periodically sprayed with water or fungi mix experienced modest amounts of corrosion. The outdoor exposure to humidity and temperature likely evaporated the sprayed solution. This depended on the season. In some cases, water was observed to be transported from the high side to the lower end of the sample. Corrosion initiated at one end of the strands for the 2-foot-long samples exposed indoors. It was observed that in some samples, the filler partially covered the strand ends. These were the samples for which corrosion progressed the most. However, it appears that corrosion was localized to the regions with a thin filler layer or no layer.

Table of Contents

List of Figures

List of Tables

Chapter 1 - Introduction and Literature Review

1.1 Introduction

The Florida Department of Transportation (FDOT) has recently implemented flexible fillers as corrosion protection for certain post-tensioning tendons (PT) used in bridge structures. The FDOT Structures Manual [1] (Structures Design Guidelines 1.11.5) describes where flexible filler is to be used. FDOT Specification 938 [2] indicates the properties of the microcrystalline wax (flexible filler). Other flexible fillers used for protection of steel tendons include greases and gels, but they are not included in the FDOT Specification at this time.

The research described in this report was carried out to better understand the flexible filler corrosion protection properties. Short (< 30 cm) and long (2 to 4-feet long) filler-coated-strand or filler-coated-wire pieces were prepared and exposed to environments that could potentially cause corrosion. A literature review was done as part of Task 1, and it is included below. Subsequent chapters describe the experiments, results (including visual observation), and forensic analysis after cleaning wire sections that were found to have experienced corrosion. The conclusions section includes a few recommendations regarding the importance of quality assurance during construction and verifying that the anchorages and other relevant sites are properly sealed.

1.2 Literature Review

Flexible filler materials and available research are discussed in depth in this chapter. There is a need to better understand how flexible fillers would perform in the Florida environment. This is important because the various filler products have different corrosion protection properties, as shown in this literature review. Installation practices also differ and may play an important role in corrosion protection. These flexible filler products are intended to be used as fillers in unbonded post-tensioning systems to fill voids and cover the steel strands to prevent corrosion of the steel from taking place. A literature review of flexible filler products used in unbonded tendons and other applications was conducted.

The review includes a brief history and how these products have evolved. Microcrystalline wax was found to be used on external unbonded tendons on bridges in France and Germany when protecting multi-strands. Other European countries like Denmark and the United Kingdom have also used flexible fillers in some bridges[3]. Flexible fillers have been used on cable-stayed bridges in Japan, Europe, the U.S., Mexico, and other countries around the world. For example, the Ravenel Bridge in South Carolina used microcrystalline wax in the anchorages.

Flexible fillers are used in the United States on other post-tensioning applications, e.g., buildings, parking garages, prestress concrete pressure vessels for the nuclear industry [4] and other structures. The flexible filler initially used in building and garage structures was a steel strand coated with grease and wrapped with paper. This was intended to be a corrosion protection prior to installation. Later, plastic tube replaced the paper. The greased strand was pushed in and was not always tight; this, in some cases, allowed for voids or steel surfaces with no grease. These two early systems have been reported to be prone to corrosion (described in more detail below). The greased strands were later encapsulated in plastic via an extrusion method[5] . Microcrystalline wax (petrolatum based) is the filler typically used in PT systems of concrete pressure vessels for the nuclear industry[6], and some manufacturers also use microcrystalline wax on mono-strands.

Prestress concrete pressure vessels (PCPV) in the nuclear industry with post-tensioning systems typically use petrolatum-based (microcrystalline wax) filler materials. However, in several publications, the words “grease” or “greased” were used where the words “microcrystalline wax” should have been used. In later sections, the original word (e.g., “grease”) found in the reviewed document is reported here, followed in parentheses by the words “microcrystalline wax” if we believe that was the investigated material. A presentation by Freyssinet [7]indicates that in other countries (e.g., India, Taiwan, Russia) greased mono-strands are used in some unbonded PT systems for concrete pressure vessels (e.g., Freyssinet VVER1000). Moreover, the properties and composition of the wax initially used in the U.S. had different properties and composition than current commercial products. In the beginning, off-the-shelf products were used instead for PCPV, which allowed corrosion to take place. There are several other countries (e.g., Japan, the U.K., France, Russia[7], [8]) that use flexible fillers in PT systems for the reinforced concrete pressure vessels as part of their nuclear power plants.

The review also looked at identifying the mechanisms to prevent corrosion of the steel strands by these flexible fillers and their possible long-term degradation mechanism. Cases in which corrosion of the steel strand took place are also part of the review.

1.2.1 Overview

Several materials have been considered to be used as non-cementitious flexible fillers such as polymers, gels, greases, waxes, etc.[9] . Some petroleum-based products that have been proposed, and used, are listed below [10], [11] .

- Gatch: mix of paraffin and waxes including a high percentage of oil (up to 30 %).
- Grease: Mix of oil and soap with anti-corrosion additives. (Note: After a time, grease can bleed, and then oil can leak).
- Microcrystalline wax: this product is obtained from heavy components of distillation. Waxes are made of saturated hydrocarbons according to formula $C_n H_{2n+2}$ types with high percentage of ramified chains and microcrystallization. This explains its flexibility, and its adhesive properties to various supports/surfaces.
- Petrolatum: a mix of paraffin and oils.
- Vaseline: result of refining and dilution of petrolatum
- Crystalline paraffin: this product is obtained by disoiling the gatch during crystallization

In Europe, for the past 20 to 30 years it was found that most suppliers were using microcrystalline waxes instead of grease because of its greater stability [12]when a flexible filler was used to fill the duct and coat multiple strands. Grease is still used in mono-strand systems, both in the U.S. and abroad.

1.2.2 Use of Flexible Fillers in the U.S.

Flexible fillers have been used to provide corrosion protection of unbonded mono-strands and multi-strand steel tendons (The reader is referred to ACI 222.2 for detail definitions for wire, strand, mono-strand, multi-strand and bar used in pre-stress or post-tension systems). Flexible fillers using grease were introduced in the U.S. during the early 1950s (ACI/ASCE 423.4R[13]) for mono-strand tendon systems. From late 1950s and 1960s the use became more common as design and materials standards were developed and established. The use of post-tensioning

increased rapidly during the late 1960s and 1970s as the advantages of the system were demonstrated for buildings and parking structures.

ACI/ASCE 423.4R report [13] describes the main advantage to be shorter construction time, reduced structural depth, increased stiffness, and savings in overall cost. ACI/ASCE 423.4R report [13]also indicates that unbonded post-tensioning systems are used in enclosed buildings, parking structures and slabs on grade. Unbonded multiwire and multistrand tendons coated with flexible filler have been used extensively in structures for the nuclear power industry, but in this application the flexible filler material used is mainly microcrystalline wax. However, in many technical papers and reports the word "grease" and "greased strands" are used when referring to the type of filler used on pre-stress concrete containers for the nuclear industry in the U.S.

Early on, the grease and sheathing were viewed by most primarily as a lubricant and bond breaker, and secondarily as a corrosion deterrent during shipping, handling, and placing. At that time, the long-term corrosion protection was viewed as being provided by the uncracked concrete cover. Subsequently inhibitors were added to the flexible filler. By the mid-1960's plastic covering started replacing paper sheathing. The greased strand was pushed-through into the plastic covering, and the fit was not always tight. The plastic covering later evolved to a longitudinal seamed plastic tube which was heat sealed. Both of these plastic sheathings had an annular space that allowed water to travel if the sheathing or anchorage was compromised. Thus, the corrosion protection of the older mono-strand systems was vulnerable at or near the anchorages. Currently, post-tension tendons are manufactured with an extruded plastic sheath tightly molded to the strand to provide superior corrosion protection. Tightly-fitting sheaths minimize voids in the grease (wax) and provide significantly less opportunity for moisture movement into and along the length of the mono-strand tendon. If a corrosion-inhibiting grease (wax) completely covers the strand, and the grease (wax) is not affected by water, corrosion generally will not occur.

The ACI/ASCE 423.4R report mentions that if water is present in the tendons enclosure, it tends to collect at low points in the profile and is more likely to be detected there, especially for loose sheathed strands [13]. When water is present, the grease can become emulsified and water then can reach the steel, which potentially could allow corrosion to initiate. Fallis and co-authors [14] describe a methodology used to evaluate if corrosion is taking place in unbonded post-tensioned mono-strand older systems (paper wrap, plastic with coated strand pushed through) by assessing the moisture content in the system. Briefly, from the Fallis paper, "The PT corrosion evaluation process works by injecting ultra-dry air under very low pressure and flow into the post-tensioned mono-strand duct at a central location. At an exit port near the end of the mono-strand, the air is directed through the PT corrosion evaluation testing unit, which verifies the flow rate and determines the moisture content of the air within the cable duct ". Fallis and his co-authors indicate that the same technique can be used to repair (by drying and applying the described system for several weeks). Ultra-dry air is blown for an extended period of time.

ACI report 222.2R [15] reports that corrosion of unbonded systems have been associated with incomplete coverage of the strands with flexible filler (grease or wax) or poor performance of the filler. Several of the available remedial options for ungrouted post-tensioned systems (222.2R [15]) are listed below:

- Delay remediation;

- Dry-gas purge and regrease the tendon ducts;
- Inject urethane into the tendon ducts;
- Strengthen the member or structure; and
- Replace the tendon.

A very comprehensive discussion of corrosion of mono-strand tendons is provided by ACI/ASCE 423.4R [13], which focus on greased mono-strands, but the discussion also applies to wax coated mono-strands or multi-strands. Corrosion problems in mono-strand systems generally result from three sources [13]:

- Sheathing problems;
- Detailing practices; and
- Storage, handling, and construction problems

The ACI/ASCE 423.4R report [13]also describe cases in which failures due to corrosion took place. The report indicates that the failure mode was identified, but it does not always mention what might have caused corrosion to initiate. For older systems in which water and/or chlorides reached the steel strands; corrosion was found in the form of pitting or uniform corrosion (at the site where water accumulated) and in some cases upon cross-section loss which allowed stress corrosion cracking to take place[16]. No specific mention of corrosion due to microbial induced corrosion is found in ACI/ASCE 423.4R. However, microbial induced corrosion (MIC) has been reported in some cases due to bacteria [17] and other cases due to fungi[18]–[23].

As indicated above, flexible fillers of petrolatum based (microcrystalline wax) type have been used in the nuclear industry. Canonico and co-authors [24] reported two incidences of grease (wax) leakage through cracks to the exterior surface of the containment apparently due to a combination of inadequate duct joints and grease (microcrystalline wax) expansion due to thermal effects. Canonico also reported that there had been two incidences of grease discoloration due to containments with water, the probable cause being entry of contaminated rain water into the tendon ducts during construction.

A more recent study by Oak Ridge National [25] Laboratory describes the leakage of the filler into concrete. The document also indicates the evolution both on composition and the type of additives added to the flexible filler for tendons used by the nuclear industry. The following sentences briefly describe how the petrolatum based filler evolved in the U.S. Initially, the flexible filler product was based on a casing filler containing polar wetting agents, rust preventative additives, microcrystalline waxes and proprietary items formulated to be water displacing, self-healing, and resistant to electrical conductivity. The next generation of materials were formed by adding a plugging agent to the flexible filler to increase the low flow point of the product (39°C (100°F)). This was done to keep the fillers from seeking loose sheathing joints and flowing into concrete hairline cracks. A subsequent requirement involved incorporation of a light base number (3 mg KOH/g of product) to provide alkalinity for improved corrosion protection. Finally, the current generation of materials have evolved through a series of modifications to produce products which have been formulated to: increase the viscosity without sacrificing pumpability, raise the congealing point to 57-63°C (135-145°F), increase the resistance to flow from sheathing joints, improve the water resistance, and raise the base number (35 mg KOH/gm product) to provide

higher reserve alkalinity. Flexible fillers have been preferred as the wires can be replaced if necessary as well as regulatory requirements for inspection[6].

Prestressed concrete pressure vessels (PCPVs) are massive structures for nuclear reactor containment. These structures involve conventional steel and a steel post-tensioning system. A study made by Oak Ridge National Laboratory [26] determined the corrosion behavior of a high strength steel in several corrosive environments and the performance of cement grout and two petroleum-based greases (microcrystalline wax) as a protection product by coating the steel strands with each one. The fillers were named type A and type B.

To evaluate the effectiveness of coating materials two different experiments were conducted: In the first one, samples were stressed to 60% of the ultimate tensile strength (UTS) while they were exposed to various corrosive solutions (for up to six days); in the second case, samples were exposed to the solutions without an applied stress (for up to 160 days) and were strained to failure afterwards. The different solutions used were: 0.1 molar solution of hydrogen sulfide (0.1M H_2S) with an approximate pH 4, a 0.2 molar solution of ammonium nitrate (0.2M $N_2H_4O_3$) and 0.1 molar sodium chloride (0.1M NaCl). The study results show that all coatings provide protection, but it's important to point out for the organic materials (petrolatum-based wax) that if a small amount of coating was removed, failures occurred in relatively short times. For the cement grout included in the study failure occurred if the flaw widths were 1.3 millimeter or greater. Griess and Naus [26] report in their conclusion that the corrosion rate of the steel was low in dilute solutions of NaCl, $NaNO_3$, Na_2SO_4 when access to oxygen was restricted, but it was substantially higher with free access to air; in the latter case, broad pits formed, even in pure water. This investigation can be considered the first long-term study to investigate the flexible filler corrosion properties (from 1,000 hours to 160 days).

In January of 1985 (NRC 2010[27]), a dented and leaking tendon greased (microcrystalline wax) cap was found during inspections at Joseph M. Farley Nuclear Power Plant; three anchor heads were found broken into pieces. Metallographic and fracture examinations showed that there was evidence of corrosion which resulted from hydrogen generation from the anodic reaction of zinc and steel in the presence of water. In 1994 several instances of higher-than expected prestressing losses, grease (microcrystalline wax) leakage through concrete, and liner bulges were reported. A more recent publication [28] contains a section that summarizes monthly operational experience reports performed at Fort Saint Vrain regarding failures of tendons. A memo from April 1984 reports that a significant number of the tendon wires used in the FSV PCPV were either corroded or failed. This was discovered during the 10-year surveillance inspection of the PCPV. The memo reports that "the licensee found broken or corroded tendon wires in the center of at least six tendons." The MR0484 (April Report) indicated that the apparent root cause of the corrosion was moisture in the sealed boxes, along with the lack of cover grease on the ends of the tendon wires. A licensee response (P-84287, dated August 20, 1984) regarding inspections and possible modifications concerning the PCPV tendon system was reviewed the following month in MR0984. It had been determined that moisture was a common element in all the cases of corrosion that were discovered, and possible microbiological corrosion was being investigated as a source or mechanism for the corrosion. A letter from the licensee (dated November, 1984) indicated that microbiological attack on the protective grease (microcrystalline wax) for the tendons was thought to be responsible for the corrosion of the tendon wires. The protective grease (No-Ox-Id an early

wax product) used was a sulfonate-based grease and was more prone to microbiological attack than an alkaline-based grease (current microcrystalline wax). It was also thought that the microbiological attack was manifested in the creation/realization of formates and acetates that were found on the wires. A memo dated February 1985 states that the corrosion mechanism was verified to be microbiological action on the grease, resulting in the formation of acetic and formic acids that led to the corrosion. The report indicates the type of corrective action pursued and that "...testing demonstrated that the tendon still exceeded the minimum acceptance criteria for design loading." The Nuclear Regulatory Commission (United States of America) keeps working in the study and improvement of greases that satisfy the standards for concrete structures in nuclear power plants [28].

1.2.3 Flexible Fillers for Filling the Ducts of Multi-Strand Tendons

Flexible filler products are intended to be used as fillers in Post-Tension (PT) tendons providing an alternative to cementitious grout in unbonded tendons. These fillers provide corrosion protection that prevents corrosion to take place on the steel stands when properly applied. The flexible filler materials have the following functions[29] :

They prevent the circulation of gases or liquids within the strand duct and in the anchorage zone.
They may provide corrosion protection and an interface
They reduce friction between metallic components and avoid fretting corrosion

Table 1 lists the requirements to meet FDOT specification (reproduces the Table in reference[2]). Table 2 and Table 3 describe the property requirement specified by the European Organization for Technical Approval (EOTA) in ETAG-013 [30] for greases and microcrystalline waxes respectively. Table 4 and Table 5 show a list of properties for grease products reported as part of previous studies regarding corrosion protection, and Table 6 shows a list of the properties reported for several crystalline wax products. Table 7 and Table 8 show the properties for some of the greases used in mono-strand systems.

Table 1. Microcrystalline wax requirements in FDOT specification [2]

Property	Test Value	Test Method
Salt Fog -168 hours at 95°F	No corrosion	ASTM B117*
Corrosive Constituent Concentration Chlorides, Sulfides and Nitrates Sulfate	 ≤ 50 ppm (total) ≤ 100 ppm	ASTM D512 & ASTM D367** ASTM D516**
Congealing Point	≥ 149°F	ASTM D938
Cone Penetration at 77°F	≤ 260 d-mm	ASTM D937
Bleeding at 104°F	≤ 0.5%	ASTM D6184
Resistance to Oxidation 100 hours at 212°F	≤ 0.03 MPa	ASTM D942
Kinematic Viscosity at 212°F	10-30 mm^2/s	ASTM D445

* Test sample consists of a 4 inch x 6 inch steel panel blast cleaned to a NACE surface preparation SP5 or equivalent, with a 2 to 2.5 mil surface profile. The plate is covered with a layer of wax equivalent to 0.5 grams wax per square inch of panel

** Prepare sample in accordance with NF M07-023, sections 6a through 6c or equivalent. Other analytical methods are acceptable as long as equivalency to the above methods has been established by the Department

Note: The microcrystalline wax used as a duct filler for post-tensioned structures, shall meet or exceed this properties at normal laboratory temperature (65°F – 78°F).

Table 2. Grease specification in [30]

Characteristics	Test method/Standard	Acceptance criteria
Cone penetration (0.1 mm)	ISO 2137	250-300
Dropping point	ISO 2176	≥ 302°F
Oil separation at 104°F	DIN 51 817	At 72 hours: ≤ 2.5% At 7 day: ≤ 4.5%
Oxidation stability	DIN 51 808	100 hours at 212°F: ≤ 0.06 MPa 1,000 hours at 212°F: ≤ 0.2 MPa
Corrosion protection: 168 hours at 95°F 168 hours at 95°F	 NFX 41-002 (salt spray) NFX 41-002 (distilled water spray)	 Pass No corrosion
Corrosion test	DIN 51 802	Grade: 0
Content of aggressive elements: Cl^-, S^{2-}, NO_3^-, SO_4^{2-}	 NFM 07-023 NFM 07-023	 50 ppm (0.005%) 100 ppm (0.010%)

Table 3. Wax specification in [30]

Characteristics	Test method/Standard	Acceptance criteria
Congealing point	NFT 60-128	≥ 149°F
Penetration (1/10 millimeter) at -4°F	NFT 60-119	No cracking
Bleeding at 104°F	BS 2000: PT121 (1982) modified	≤ 0.5%
Resistance to oxidation 100 hours at 212°F	AST D942.70	≤ 0.03 MPa
Copper-strip corrosion 100 hours at 212°F	ISO 2160	Class: 1a
Corrosion protection: 168 hours at 95°F 168 hours at 95°F	 NFX 41-002 (salt spray) NFX 41-002 (distilled water spray)	 Pass No corrosion
Content of aggressive elements: Cl^-, S^{2-}, NO_3^-, SO_4^{2-}	 NFM 07-023 NFM 07-023	 50 ppm (0.005%) 100 ppm (0.010%)

Table 4. Properties of grease used by De Leo [23]**.**

Characteristics	OVOLINE 71C
Soap type	Calcium
Soap content	11% Approx
Worked penetration at 77°F	270
Drop point	149
NLGI grade	2
Appearance	Amber
Texture	Smooth
Free alkalinity (IP 37)	0.2% max
Copper corrosion (IP 112)	Pass
Oxidation stability (ASTM D942)	9.2 psi.

Table 5. Properties of grease used by Mietz et al. [31]

Properties	Standard	Texaco Lithiac 142 MP
Color	__	Brown
Thickener type	__	Lithium
Viscosity (mm^2 /s): 104°F 212°F	DIN 51 562	 612 – 748 25
Penetration worked (mm/10)	DIN ISO 2137	330
Dropping point (°F)	Mettler	> 320
Water Spray-Off, % weight	ASTM D 4049	< 2.0
Rust protection	ASTM D 1743	Pass

Table 6. Properties of waxes used by Mietz et al. [31]

Properties	Standard	Denso-Jet
Color	--	Dark brown
Density at 73.4°F (g/m^3)	ISO 2811	0.94
Dripping point (°F)	DIN 51801	154.4
Viscosity (mPa s): 131°F 149°F 185°F	DIN 53019-1	 -- 2,000 450
Water absorption (% weight) 1 day 23 days	DIN EN ISO 62	 <0.01 0.08
Saponification value (mg KOH/g)	DIN EN 12068	1.0
Volume resistivity (Ω cm)	DIN IEC 60093	$> 10^9$

Table 7. Other grease products found [32]–[35]

Characteristics/Properties	NO-OX-ID "AC"	Red-I PT	Specis B 4851/2	Visconorust 2090 P-4
Color	Dark Amber	Amber	Brown	__
Soap Type	__	Lithium	Lithium	__
Dropping Point (°F)	130 -170 (ASTM D 127)	383 (ASTM D 2265)	>365 IP396/ISO2176	__
Pour Point (°F)	130-160 (ASTM D 97)			135 (ASTM D938)
Cone Penetration at 77°F	110-170 (ASTM D 937)	265-295 (ASTM worked)	245-275 (ASTM D217)	170 – 200 (ASTM D937)
Rust Test	__	Pass (ASTM D 1743)	__	__
Corrosion Test	__	Pass (No Rust) (ASTM B 117)	Pass (No Rust) NF X 41-002	__
Soak Test	__	Pass (ASTM B117)	__	__
Flash point °F	400 (ASTM D 937)	350 (ASTM D92)	__	420 (ASTM D92)
Base oil viscosity: At 40°C (cSt) At 100°C (cSt) At 100°F (SUS) At 210°F (SUS)	 __ __ __ __	(ASTM D88) 321 21 74 130-300	__	 __ __ __ __
Content of aggressive elements: Chlorides (ppm) Nitrates (ppm)	 __ __	 < 0.5 (ASTM D 512) < 0.1 (ASTM D3867)	__	 < 2.0 (ASTM D 512) < 4.0 (ASTM D3867)

Table 8. Other wax products found[36].

Characteristics/Properties	Standard	Anticorit 28890
Color	__	Tan
Consistency	__	Soft pliable, self healing thixotropic
Specific Gravity	ASTM D-1475	1.00-1.05
Density @ 60°F (15.5°C)		8.3-8.7 lbs/gal
Drop Melting Point*	ASTM D-2265	500°F (260°C)
Rust Test	ASTM D-1743	Pass
Corrosion Test	ASTM B-117	Pass (No Rust)
Soak Test	ASTM B-117	Pass
Flash point °F	ASTM D-92	347°F (175°C)
Base Number	ASTM D-974	170-230 mg KOH/g
Base oil viscosity: At 77°F 25°C (cPs)	ASTM D-2196	120,000-150,000
Content of aggressive elements: Chlorides (ppm) Nitrates (ppm) Sulfides (ppm)	ASTM D-512 ASTM D-3867 Hach or APHA 4500S2	10 ppm max. 10 ppm max. 10 ppm max.

1.2.4 Bridges That Have Used Flexible Filler on Unbonded PT Systems

Germany and France's current practices are to use flexible fillers for external tendons. Microcrystalline wax material is thought to have a good stability at high temperatures. The European Organization for Technical Approval (EOTG) in ETAG-013 [30] have a minimum of required criteria to consider an acceptable wax based on material stability, separation, penetration, aggressive ion content, and corrosion protection.

Godart's paper [37] describes that the Villeneuve Saint-Georges Bridge (built in 1953) presented an abnormal vibration of certain tendons during the passage of heavy vehicles in 1978/79, and as a result of this, some wires broke. A favorable atmosphere for corrosion existed within the box girders because the access points for inspection that were located in the upper slab were not tight, and they allowed water to filter in. After an inspection in 1980, a questionable tendon was replaced, and all tendons were re-tensioned to their initial tension, and grease was applied to re-protect all tendons. In this case, flexible filler was used as the original filler and as a repair methodology. The study described other bridges with external tendons and showed that the durability of tendons well coated with grease was assured, provided that these tendons were in well ventilated box girders (i.e., good drainage so that water did not accumulate at the anchorage). Thus, if the filler-coated wires are in a wet atmosphere, failures caused by corrosion might occur, and the durability of the tendons could then be compromised.

France is the largest user of wax-filled tendons. In 2001, the LCPC (*Laboratoire Central des Ponts et Chauss*ées) [11,39] presented a study about the status of durability of post-tensioned tendons in France. A paragraph in the paper talks about flexible fillers (soft products) and mentions that

external tendons (multi-strand) injected with grease did not provide satisfactory results because the grease proved to be unstable and leakage occurred due to non-tight ducts (this took place at the Bayonne Bridge). A publication by Virlogeux [38] indicates that flexible fillers (grease injection) were first used as repair method on external cable stays: e.g., temporary strengthening of the Tours Bridge, strengthening of the Bayonne and Layrac Bridges. The La Fleche Bridge was erected using grease injection. The external tendons of the Vallon des Fleurs and La Banquiere viaducts were thus injected in plastic ducts, with grease/wax heated to about 50°C (i.e., fillers with low viscosity at relatively low temperatures)[38]. The paper by Godart [37] mentions that now soft injections are carried out with crystalline wax which are more stable products. Godart states that, so far, microcrystalline wax material has not shown any durability issues, based on the few sheaths or anchoring caps openings which have been made[37].

After decades of successful bridge design with external post-tensioning, Germany has moved towards using unbounded tendons internally, as well. The friction coefficient of prefabricated tendons with wax as corrosion protection is significantly smaller than the one of bonded tendons with metal ducts; this can be of special interest in tanks and multi span bridges where large deflection needs to be done. The pilot project was the Kleine-Laaber-Viaduct in South Germany, a 273 m long structure. With vacuum help the duct has been filled nearly completely: up to 99.9%. Inspection of the cables at the supports has shown that there was no entrapped air. Finally, high quality corrosion protection and the handling of replacements were achieved[39].

Table 9. Post-tensioned bridges in the UK.

Bridge	Date Opened	Corrosion Protection
Braidley Road	1970	Wires coated with Brindon's Metal Coat A and each strand in a PVC sleeve
Canning Town	1972	Strands individually sheathed in grease filled PVC sleeves grouted at anchorages
A3/A31	1976	Each strand in grease filled PVC sheath, grouted at anchorages
Robsart Bridge	1977	Each strand in a grease filled polypropylene sheath, grouted at anchorages
Exminster Viaduct	1977	Each strand is grease filled polypropylene sheath.
Basingstoke Canal Aqueduct	1995	Individually sheathed mono-strands in wax filled HDPE ducts
Batheaston – Avon Viaducts	1996	HDPE ducts filled with grout, wax at anchorages
Second Severn Crossing	1996	HDPE ducts filled with wax.
A13 Flyover	2000	HDPE ducts filled with grout.

During the period of 1970 to 2000, the United Kingdom built 10 bridges (See Table 9) with either grease- or wax-filled tendons during the moratorium on cementitious grout[40]. Woodward describes the performance of post-tensioning tendons in bridges in the UK. After the collapse of a small bridge at Ynys-y-Gwas in South Walles, caused by corrosion of PT strands passing unprotected through porous mortar joints, an investigation was initiated into the durability of bridges. A program of post-tensioned concrete bridge special inspections (PTSI) undertaken in the United Kingdom showed that the condition of post-tensioning systems in most of the cases was satisfactory although many contained voids in the ducts and a light presence of corrosion was found in some tendons[40].

Fuzier and Chabert [10] reported two field cases that used flexible filler (microcrystalline wax) in cable stays: one in Tampico, Mexico, and a second bridge in Elorn France. In the latter, galvanized steel strands were threaded and tensioned strand by strand. Afterward, the wax injection was done after ensuring the perfect tightness of the duct.

Zilch, et al., [41] described future trends in Germany for unbonded tendons: the paper stated that most box girder concrete bridges are post-tensioned with both external tendons and bonded internal tendons that are arranged in the slabs of the superstructure. A future trend in Germany according to Zilch might be full unbonded post-tensioning: meaning using unbonded rather than bonded internal tendons. A couple of examples of fully unbonded post-tensioned bridges are given by Zilch, et al. Mühlenbergbridge, North Rhine-Westphalia and Roßriether Graben-Bridge, Bavaria.[41].

Zilch, et al., mentioned that there are some different requirements that unbonded tendons for internal use have to fulfill in contrast to external use[41]. The high density polyethelene (HDPE) sheathings are covered with concrete for the whole length. Due to the bond between polyethylene and concrete friction occurs between polyethylene and prestressing steel during stressing. This kind of friction might wear the pipes. Since the tendons are arranged within the concrete section in the slabs they are reducing the shear capacity of those members. Therefore, according to Zilch, a future trend is to reduce the size of the tendon cross-section. Several mono-strands can be arranged in a very compact way (Fig. 11 in Ref [41]reproduced below as Figure 1). For a tendon using 16 mono-strands with a single sheathing 45 % of the tendon cross-section is filled by prestressing steel. When using ordinary unbonded (mono-strand) tendons, this rate is about 25-30 %.

Figure 1. From Zilch et al. Left : Mono-strand and corrosion protection mass coated with a single or double polyethylene sheathing (PE-HD); Right: Assembling of sixteen mono-strands to a larger tendon [42], [43]**.**

1.2.5 Field Examples Where Corrosion took Place on Wax-injected Systems

Recently, Gilles [44] reported a case in which corrosion took place at the anchorage for microcrystalline injected tendon systems. The external post-tensioning was renewed in 1991 with wax sheathed mono-strands on a cantilever box girder bridge built in 1951. In the transversal beam, at the cantilever girder hinge, post-tensioning became internal up to the anchorage. Strands were placed in a plastic duct with wax injection. In 2005, sheathed strand ruptures were observed in the box. The inspection revealed that the problem originated at anchorages in the cantilever hinge. Gilles reports that at the downward tendons, several liters of water were locked in the anchorage head. The injected wax floated over the water. This environment allowed for the development of stress corrosion cracking of the strand just behind the anchorage head.

Ebeling et al. [45], reports corrosion of some strands on multi-strand anchor tendon system also at the anchorage. Two deteriorated anchor heads were opened; the report describes that the flexible filler appeared highly contaminated (the report indicates grease, but appearance in pictures are of microcrystalline wax). In the upper section, it was red from rust products and, in the lower section, water and possible mildew had turned it black.

Robson and Brooman [46] report corrosion-related distress in a precast segmental box-girder bridge with external tendons (1997). The external prestress was provided by 240 tendons, each consisting of 19 wires 19 millimeter (3/4 in.) diameter inside a plastic, grease-filled sheath. Severe signs of distress were observed after approximately 20 years of service. Two of the 240 tendons had failed completely, and evidence of individual wire fracture was observed in 121 of the

remaining tendons. The fractures were attributed to corrosion of the wires in the anchorage zones. Robson and Brooman reports that it was assumed that corrosion began during a 10 month construction delay, during which the tendon ends were left unprotected. Because the tendons were external, individual wire failures were detected by visual inspection. Existing tendons were removed, and the bridge was prestressed with new tendons after modifications to the anchorage areas.

1.2.6 Corrosion of Steel Coated with Flexible Fillers

Corrosion of steel coated with flexible fillers has been observed due to the following reasons.

1) Water presence (either due to holes/voids and/or not properly sealed) caused corrosion on early (grease) types of flexible filler and sheathing. In some cases, chlorides also contributed (either a higher concentration than specified was present in the filler or chlorides migrated through the concrete and reached the strands coated with grease and paper sheathing).
2) Poor quality of the flexible filler application (when an early-on manual application was used) and/or poor quality of the filler
3) Microbial influenced corrosion due to bacteria
4) Microbial influenced corrosion due to fungi

Unbonded post-tensioned tendons may corrode for a number of reasons, but primarily due to voids in the protective grease/wax, which allows moisture to accumulate adjacent to the post-tensioned tendon. Some of the situations where corrosion of these cables might appear are: unprotected structures (such as parking garages exposed to the weather), where cable wetness and cable corrosion have been identified due to leakage from unsealed anchorages (this could be due to either or both leakage of filler at anchorage which allows water and contaminants to sip into an unsealed anchorage); and/or buildings in hot, humid environments, where cool air-conditioned interiors can facilitate elevated humidity on interior cables due to climatic difference [14].

Some of the types of corrosion [14] in unbonded post-tensioned mono-strands are: Uniform corrosion; pitting corrosion, the formation of an electrochemical cell causing localized corrosion which allows brittle failure after a negligible loss of metal; stress corrosion (the combination of high-tensile stress and corrosion which can cause corrosion cracking in post-tensioned wires); and hydrogen embrittlement (when hydrogen has the opportunity to penetrate the wires and recombine with hydrogen molecules, causing an internal pressure in the metal, that could cause fracture at lower stress).

1.2.6 Short- and Long-Term Studies that have Assessed Corrosion Protection

Jean-Philippe Fuzier and Alain Chabert [10] presented some developments about petroleum wax products used as corrosion protection. The Fuzier and Chabert paper also includes a summary of the results for tests performed on twelve products (no table was provided in the paper, but it appears that both microcrystalline wax and grease products were tested). Different tests and test durations were carried out. The summary states that tests lasted 6 months, 9 months and for another series 31 months (with one month interruption during month 22) [The original reference by Chabert and Creton was not obtained]. The thickness of the filler for some samples was very thin (Transparent layer and < 0.2 millimeter), but for others the thickness ranged from 0.5 to 1 millimeter. The

mechanical characteristics of the non-protected steel was determined by tensile tests before and after the stress corrosion. A series of comparative stress corrosion tests were carried out in distilled water. Samples with different layers of protection product were put in waterproof cells and then installed on constant length devices. A load was applied. Even though some protection products, such as complex aluminum grease and calcium grease increase the life span of the strands, the study demonstrated better behavior of petroleum wax products. The tests lasted 31 months without rupture or loss of mechanical characteristics.[10] report illustrate that petroleum wax protection products increase the life span of the strands in the standardized stress-corrosion test (NF A 05-302) compared to non-coated strands.

Mietz et al. [31] investigated the long-term corrosion protection behavior of flexible filler materials, three grease and three wax products were tested. The exposure tests had 1 millimeter or 5 millimeter layer of different commercially available corrosion protection materials on top of steel wires and were exposed to different conditions. One experiment consisted of exposing the fillers to condensed water (100% relative humidity at 30°C). Filler-coated steel strands were exposed to simulated soil conditions that contained microbiological contaminants (as per DIN EN ISO 146 and DIN 50929-3), and an artificial soil solution with pH 4.5 (5 millimeterol Cl^-/kg and 50 millimeterol SO_4^{2-}/kg) with no microbiological contaminants. After certain periods of time, selected samples were taken out from the environmental chamber for the samples exposed to condensed water and the filler was removed for inspection. The evaluation was carried out after 200, 300, 400, and 500 days on selected samples exposed to simulated soils, soil or solution. Both visual inspection (regarding possible corrosion attack) and the mass losses were determined by gravimetric measurements on the selected samples exposed to simulated soils. More severe corrosion effects were observed on steel exposed to simulated soil that contained microbiological contaminants when compared to the specimens exposed to the condensed water or the artificial soil solution with no microbiological contaminants [31].

A fourth test consisted of extended time tests with stressed prestressing steel wires under soil conditions as per DIN 50929-3 and the artificial soil solution described above. A single steel wire was tensile stressed to 0.75 Rm. For samples exposed to condensed water and greased (i.e., a thin layer of wax/grease that was not uniform) significant corrosion attack took place. For those coated with one millimeter of flexible filler (exposed to condensed water) inspected at 180 days, the mass loss ranged between 0.003 g and 0.01 g; after 500 days, the mass loss ranged between 0.022 g and 0.033 g and depended on the type of filler. Samples exposed to artificial soil solution (no micro-organisms) showed lower corrosion rates for the specimens with wax-like compounds compared to the oil-base products. After 500 days, a significant increase of the mass loss was determined for the wax-like materials. The higher corrosion was observed on steel coated with fillers and exposed to very aggressive soil with microbiological contaminants, regardless of the filler. The absolute mass loss values were up to 100 times higher than those exposed to condensed water. In the exposure under soil conditions with direct soil contact, the wax-like compounds showed a better behavior than the greases. Mietz et al. [31] reports that this is due to the different consistencies of the materials. The wax-based materials showed a higher resistance against the ingress of substances of the soil and yielded better protection properties under these conditions.

Mietz research group evaluated the water uptake of the flexible fillers that were investigated. The test was carried out for 210 days and ranked the materials. Slightly better corrosion protection was

found for two of the wax fillers (< 0.5 % mass gain after 210 day) than the greases based on oils containing metal soap (1% to 1.5% mass gain after 210 days). However, the third wax filler had the largest water-uptake (2% by day 120).

Parallel to the long-term exposure test two electrochemical tests were performed: 1) impedance measurements and 2) measurement of galvanic currents were evaluated for corrosion protection and water absorption of the fillers on a special cell. For the latter test, one copper rod, one magnesium rod and two steel wires were used as electrodes. The electrochemical measurements are based on the registration of sub-microscopic electrochemical reactions at the used electrode sensor instead of a macroscopic corrosion effect on the steel specimen. The results show that most of the protection materials have significant changes between 100 and 500 days as measured by impedance. Flexible filler was applied with a 1 millimeter layer on a steel wire and the reduction in impedance monitored from 1 to 10 GΩ to when it went below 10 MΩ. Two of the greases experienced such reduction at 100 and 150 days; one microcrystalline wax product experienced the decay by day 300 and the third grease product by day 450. The other two microcrystalline wax products did not experience a reduction in impedance below 10 MΩ. The special cell used to measure small galvanic current (initially < 10 pA with no degradation) had a 1 millimeter layer of filler and water on top of it. The water uptake reduces the filler resistivity and an increase in electrochemical activity allows for a larger current to be measured. The increasing current is correlated with a progressing reduction of the protection properties of the coating. The contact currents between steel and magnesium showed a higher increase (>500 pA) compared to the current between steel and cooper (~100 pA) after 60 days [31] for rods coated with a grease product, no change was observed after 120 days on the sample coated with microcrystalline wax.

1.2.7 Microbiologically Influenced Corrosion (MIC)

A few publications indicated that corrosion of steel strands was likely due to microbial- induced corrosion (MIC). Microbiologically induced corrosion is caused by the presence and activities of microorganisms. MIC is best understood as microbiologically mediated reactions with metal oxides, protective material layers, or the formation and dissolution of minerals [47]. In this type of corrosion, different bacteria or fungi can develop that later produce an acidic environment that allows corrosion to initiate.

When focusing on the role of fungi, it has been seen that fungi are frequently involved in the bio-deterioration of materials such as polymers, paints and organic coating such as greases and microcrystalline waxes [48].

Toropova et al. [49] determined that 80% of lubricants used for protecting materials were contaminated with 37 biological agents (21 fungi and 17 bacteria) during storage and use, independent of climate or relative humidity. They identified the following species as the most frequently encountered: *Penicillium chrysogenum*, *Penicillium verrucosum*, *Aspergillus versicolor,* and *Bacillus pumilis* [49]. The growth of any microbial specimen in lubricants was accompanied by changes in color, turbidity, acid number and viscosity, where acid number refers to the base or acid composition of the compound [22].

One method of protecting some of the flexible fillers is the addition of biocides to prevent MIC. Some of the most used and effective additives are tributyltin oxide (TBTO) and alkylphenoxytriethyl-stannane (Afotas). These additives should not have a detrimental effect on the physicochemical and operational properties of the fillers and should be non-toxic [49] for humans. Some researchers [18]–[23] warn about verifying the compatibility of the filler, steel, and the environment.

There is evidence based on reported case studies that corrosion has been observed in mono-strand systems coated with flexible fillers. In some cases, corrosion of the steel was followed by stress corrosion cracking (SCC) failure. Corrosion, in some instances was initiated by microbial induced corrosion (either bacteria or fungi, whereas in other cases, the reason for MIC was not investigated).As corrosion propagated, it evolved into SCC. Regardless of the source of MIC, an acidification of the wax/grease took place (sometimes identified via pH paper, or by the smell of the acid). De Leo [23] mentions two references ([52,][50] which were not written in English) and Little mentions two references ([6] and a NRC Engineering report). Little reports that investigators for NRC concluded that the microbiological breakdown of organic grease (microcrystalline wax) resulted in the formation of formic and acetic acids which combined with moisture and thus caused corrosion. The work by Little and co-workers [18-22] provided the first investigation and evidence of the role of fungi in the degradation and corrosion processes of lubricated sheathed carbon steel tendons. A similar study was later carried out by De Leo's group [23].

MIC case studies that involve bacteria

Little's investigation reports [18]–[22] previous research carried by others that found bacteria as the source of corrosion for steel coated with flexible fillers. For example, MIC has been documented for prestressed tendons in a concrete reactor vessel at Fort St. Vrain Generating Station, Denver, CO [17][28]. Ashar et al. [17] concluded that the microbiological breakdown of organic grease (microcrystalline wax) resulted in the formation of formic and acetic acids which combined with moisture and caused corrosion of the steel. Corrosion was observed in areas where grease had been consumed or removed during placement of the tendons.

MIC cases studies that involve fungi

Little and Ray [18] published a conference paper that described the different materials in which fungi can grow and fungi's ability to remain in dormant stage. Little, et al. [19]–[21], realized a study of fungal-influenced corrosion in post-tension structures where different laboratory experiments were accomplished in order to demonstrate that fungal degradation of greases can produce organic acids and lead to corrosion of carbon steel cables in polyvinyl chloride sheaths [20], [21]. Fungi were isolated from the lubricating grease that covered a steel strand. The grease was found to be acidic as measured with pH indicating paper. Three different species of fungi were used in this testing: *Fusarium sp, Penicillium sp*, and *Hormoconis sp*. Fourier transform infrared spectroscopy (FTIR) was used to determine the acid production and the grease degradation. In addition, a scanning electron microscope (SEM) was used to observe the association between the fungi with corrosion products and the details of the corrosion [20]–[22].

Samples from corroded areas in carbon steel cables were inoculated in a Petri dish with grease and showed the positive presence of *Fusarium sp.* and *Hormocomis sp*. Different Fourier transform infrared spectroscopy spectra were collected from the contaminated coated strand, and demonstrated different degrees of degradation. As the grease oxidized, the carboxylate contribution decreased and the carbonyl contribution increased [20]–[22] . Carbonyl compounds originated by the reaction/combination of carbon monoxide and the metal that was corroding. The rate of carbonyl increased proportionally with the carbon monoxide present in the environment; carbonyl formation also increased with the content of hydrogen, ammonia and sulfurous [51]compounds that can be produced in the interaction between the fungi and the filler.

De Leo, et al. [23] showed that even though microbial influenced corrosion is primarily due to bacteria, fungi can be the culprit as well. Four fungi: *Aspergillus falvus*, *Penicillium commune*, *Fusarium solani*, and *Acidomyces acidophilum* were studied on a commercial calcium based grease (OVELINE 71C) that is used to provide protection against stress corrosion in unbounded post-tensioned coated tendons. This study used SEM and FTIR spectra for organic acid produced by fungi. The results of different tests showed that all four fungi utilized the grease as a carbon source. The grease texture changed as a result of fragmenting by hyphae penetration of fungal colonization. The grease also became thinner. Fungi spores exist in all environments, but may be additionally present in post-tension strands if they have contaminated the grease at any stage. Fungi grow rapidly as soon as water reaches the dormant structures; most of the fungi species only need a carbon source and water. As long as hydrocarbons are available, fungi will grow [23]. De Leo [23] states that fungi metabolically adapts to all sorts of climate and environmental stresses in order to germinate and colonize which in turn can create structural failure in reinforced concrete with PT systems due to the release of organic acids and the production of hyphae (when in flexible fillers coating steel strands). Fungi generally need a nitrogen source in order to grow, but in the experimental protocol, they were not supplied with nitrogen and yet still grew. This means that fungi will find any necessary means to support their growth in terrestrial and subaerial environments. It is speculated that MIC due to fungi could also take place on steel strands coated with contaminated microcrystalline was. This will be investigated as part of the experimental component of this project.

1.2.8 Non-destructive Testing (NDT)

Matt Peter [52] presented a brief discussion of the inspection and maintenance of post-tensioned tendons in existing and future prestressed concrete structures. The inspection methods theoretically available can be distinguished between non-destructive and destructive methods. With the non-destructive methods, it is not possible to check the actual mechanical properties directly, but it is possible to measure some auxiliary values which might be related with what the responsible engineer wants to know and show the changes of these values and some properties over time [52].

The International Federation for Structural Concrete presented in their Bulletin No.33 some non-destructive testing for the inspection of post-tensioning tendons [11]. Some of the NDT listed are georadar, covermeter, fiberscopy, potential mapping, radiography, ultrasonic methods, acoustic monitoring, thermography, and tomography. The FIB bulletin presented an acoustic monitoring in a bridge in Roveredo, Switzerland [11].This technique has potential for monitoring deterioration

in unbonded strands, but is ineffective for bonded strands because of severe attenuation of elastic waves due to the surrounding concrete or grout [11].

Ebeling et al. [45] affirmed that the most comprehensive effort related to the NDT of post-tensioning was found in the National Cooperative Highway Research Program NCHRP (1999). The nonlinear vibro-acoustic testing method has potential for long-term success in providing information with respect to the condition and soundness of the concrete-steel interface. This acoustic testing method cannot provide a direct measure of cross-sectional area loss or the presence of cracks in the strands; this technique is not applicable to grouted cables [45]. The method with the highest potential for detecting flaws in post-tensioning tendons is the magnetic flux leakage. In this method, two large neodymium iron boron magnets are used. It is effective when there is close proximity between the sensors and the wires (a few inches), which makes this approach useful for near surfaces cables [45].

The Highways agency in the UK realized a five-year special inspection program [53] using NDT methods in order to detect voids and damage in post-tensioned bridges. Some of the techniques studied at the Transport Research Laboratory (TRL) were: 1.) Radiography (x-ray with a 3-curie Co-60 source) which detected voids but could not detect corrosion and 2.) Impulse radar at 900 MHz and 1 GHz, using analog and digital systems. The impulse radar method could not see beyond metal ducts, but can be used to detect metal which is reliable to detect voids in ducts. Techniques such as ultrasound, and tendon detectors were partially successful. Electrical reflectometry tests were disappointing. All NDT methods are susceptible to misapplication and misinterpretation [54]. Additional NDT are described in references [55], [56].

Chapter 2 - Experimental

In the literature review, we reviewed a variety of different laboratory tests done by others to investigate the performance of flexible fillers. Some of the experiments performed as part of this investigation described in this chapter were inspired by the tests described in the literature review. In addition, larger samples were prepared in which the flexible filler was injected. Most of the larger samples were exposed outdoors.

The aim of the tests was to characterize the corrosion protection of flexible fillers (microcrystalline wax materials). A total of five fillers were investigated: Trenton, Civetea, Garringer (Nontribos), Sanchem, and Sonneborn.

Larger samples were prepared by injecting the flexible filler into 2-inch diameter tubes (either polycarbonate or high density polyethylene), the preparation of these samples is described in detail later in this chapter. These samples were 2-feet long or 4-feet long.

Smaller samples tested involved exposure of filler-coated strands/wires to high moisture or outdoor environment, whereas other tests exposed the filler by itself. The filler-coated strands were obtained from tendon segments removed from a mock setup prepared at FDOT-SRC, and the filler was injected.

The flexible filler was applied manually on the single-wire tests or by a modified method described below. Three different fungi species were introduced purposely (by spraying each of the selected fungi or a fungi mix) to contaminate the fillers mentioned above. Two types of steel specimens were used in the experiments: Seven-wire strand specimens and single-wire specimens. Some of these specimens were coated with flexible filler and then exposed to fungi.

Another experiment consisted of outdoor exposure of as-received flexible fillers and another set of flexible filler samples were exposed in the lab after inoculation with fungi.

2.1 Materials

2.1.1 Flexible Fillers

Five corrosion protection materials were used (microcrystalline waxes):

- Trenton
- Civetea (Cirinject CP)
- Gärringer (Nontribos VZ Inject)
- Sanchem (NO-OX-ID-NG)
- Sonneborn (Visconorust-2090-P)

The properties of these flexible fillers are listed in the Table 10. All of these materials are microcrystalline waxes.

2.1.2 Fungi Species

Three different species of fungi were chosen for the experiments: *Fusarium oxysporum*, *Penicillium chrysogenum,* and *Aspergillus flavus*. *Fusarium oxysporum* is a genetically heterogeneous polytypic morphospecies whose strains represent some of the most abundant and widespread microbes of the global soil microflora (Fig. 2a) [57], [58][59]. *Penicillium*

chrysogenum is a species of fungus and common in temperate and subtropical regions (Fig. 2b) [60]. It can be found on salted food products, but it is mostly found in indoor environments, especially in damp or water-damaged buildings. *Aspergillus flavus* is a saprotrophic and pathogenic fungus with a cosmopolitan distribution (Fig. 2c) [61][62],. These species were recently investigated and shown to cause corrosion of flexible filler-coated steel wires [23]. All fungal colonies were prepared from actively growing cultures and were grown and maintained on potato dextrose agar (PDA; Oxid, Inc) in Petri dishes (3.5 inches or 4 inches diameter) at 22°C and laboratory RH. The petri dishes were kept in the Marine Materials and Corrosion Laboratory at Sea Tech at room temperature and laboratory humidity (65 to 70% Relative Humidity).

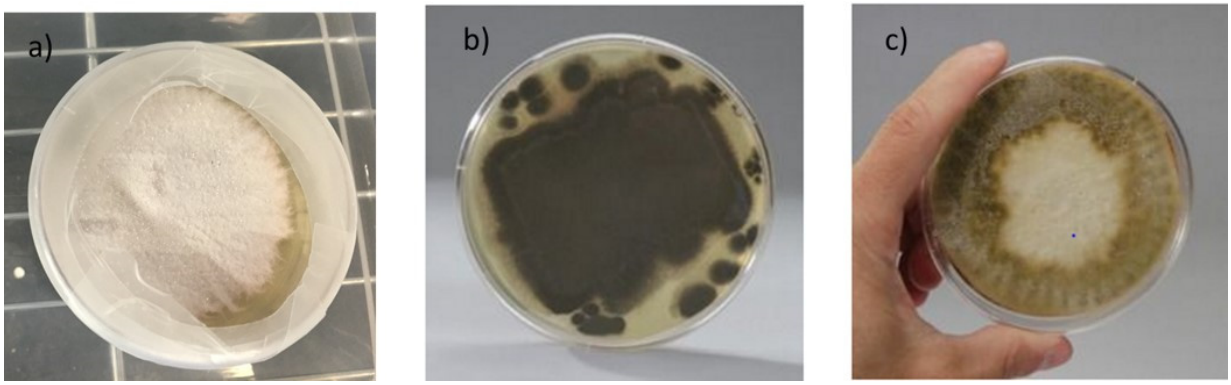


Figure 2. Fungi species: (a) *Fusarium oxysporum*, (b) *Penicillium chrysogenum*, and (c) *Aspergillus flavus* [1].

2.1.3 Seven-wire Steel Strands

Seven-wire steel strands with a length of at least 4 feet coated with three different types of filler were provided by FDOT-SRC. Coated strand segments were given to FAU during a visit to the structures lab that took place in March 2016. These strands were coated with Civetea and Sanchem. These coated strands were part of the initial mock-set-up prepared for a FDOT project with UF. The third type of coated strands (also part of a mock set-up) were provided via cut off tendon segments during a visit to FDOT-SRC that took place in mid-October 2016. These strands were coated with Trenton. Some of the segments were selected for the lab experiment and removed from the HDPE duct enclosure. These pieces were cut into 4 feet long segments.

During the visit to the Structures lab in March 2016, uncoated strands were also provided. One of these strands was cut to obtain smaller pieces for use either as single-wire or seven-wire smaller/shorter segments.

[1] Figure 1 a. FO in a petri dish in the Marine and Corrosion Lab. Figures 1b and 1c from the Carolina Biological website: https://www.carolina.com/fungi/penicillium-chrysogenum-living-plate/156146.pr?question=pennicillium+chrysogenum#

Table 10. Properties of candidate flexible fillers [63]

Physical Properties	Sanchem (NO-OX-ID-NG)[2]	Sonneborn (Visconorust-9090-P) [1]	Gähringer (Nontribos VZ inject) [1]	Civetea (Cirinject CP) [1]	Trenton	Lubriplate (No. 133-AA)
Density (g/cm^3)	—	758-870	—	880	—	—
Specific Gravity at 60°F	0.88-0.95	0.88-0.94	—	—	0.80-0.90	0.94-0.96
Drop melting point °F	135-155	35-80	80	65-75	80-100	190 - 199
Congealing Point °F	—	135	68	—	45	—
Flash Point °F	420	420	>536	392	300	399.2
Cone Penetration at 77°F (1/10 millimeter)	160-250	170-200	85	65-115	—	—
Viscosity at 210°F (mm^2/sec)	—	5-30	20-25	17-22	—	135 (at 104°F)
Solubility in water	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble	Insoluble
Salt spray test (168 hours at 95°F)	No corrosion	—	—	No corrosion	—	—
Copper corrosion	—	—	No corrosion	No corrosion	—	—
Water soluble Chlorides	< 5 ppm	2 ppm	40 ppm	—	—	—
Water soluble Nitrates	—	4 ppm	10 ppm	—	—	—
Water soluble Sulfides	< 2 ppm	2 ppm	10 ppm	—	—	—

[2] Taken from Table 2. Properties of different injection materials.[63]

2.2 Filler Deterioration Tests

2.2.1 Behavior of Flexible Filler

To assess the environmental effects on the flexible fillers a certain amount of each filler was placed in mid-size plastic containers with no cover (the containers were about a shoe-box size). A set of containers, two replicas per filler were exposed to two different environmental conditions. Both environmental conditions were located outdoors at Sea Tech:

1) A pair of large partially closed containers (one is 29 inches × 30 inches × 17 inches) stored the small containers with the flexible fillers. The humidity was kept high inside the partially closed container by adding water to the bottom of the container. The large bin was not hermetically sealed and particulates were able to enter during inspection and if the day was very windy; (see pictures in Figure 3.(a))

2) A second set of small bins containing the different fillers was placed on a metallic shelf on the second floor at a partially sheltered location. This type of set-up was developed in order to see how long it takes the samples to get contaminated by airborne particulates. Continuous visual inspection was performed regarding the contamination (determined by appearance). (see Figure 3.(b))

Figure 3. (a) Containers outdoors, (b) Metallic shelf outdoors.

2.2.2 Temperature Effect on Inoculated Fillers

Suspensions were prepared from young, actively growing fungi cultures. After the fungi had grown for 20 days at 22°C, fungal colonies were scraped from the agar surface and suspended in distilled water. Petri dishes (2.2 inches diameter) filled with each of the different fillers were then inoculated with each of the three different fungal colonies: *Fusarium oxysporum*, *Penicillium chrysogenum,* and *Aspergillus flavus*. A total of 20 samples, 4 per each filler, including a negative control without inoculation, were left in the laboratory at 22°C. The goal was to check if the fungal colonies grew in the flexible fillers and assess how long it took to spread. Monitoring of fungal growth in the microcrystalline waxes was performed by periodic visual inspection of the Petri dishes.

A second set of Petri dishes (3.5 inches in diameter) were filled with each of the different fillers and inoculated with each of the three different fungal colonies: *Fusarium oxysporum*, *Penicillium chrysogenum* and *Aspergillus flavus*. A total of 20 samples, 4 per each filler, including a negative control without inoculation were left in an elevated temperature room at 32°C to check if the fungal colonies grew in the flexible fillers and assess how long it took to spread. Monitoring of fungal growth in the microcrystalline waxes was performed by periodic visual inspection of the Petri dishes.

2.3 Coated Strand/Wire Exposure Tests

2.3.1 Filler-coated Strands

A set of strands with a length of approx. 4 feet coated with three flexible fillers (Civetea, Nontribos, and Trenton) were provided by SRC-FDOT. The coated strands were exposed to two different environmental conditions, both of which are located outdoors at Sea Tech: 1. Coated strands were placed in an enclosed container (samples were exposed to this condition for 353 days); 2. Coated strands were placed outdoors, at a partially sheltered (see Figure 3(b)) location.

In both conditions, the strands were not supported directly to a base. A space was kept between the base and the strand in order to have a homogenous air flow on both sides of the strand (except at support points). With this type of set-up, it was possible to determine when corrosion initiated on the strands and how it evolved. Continuous visual inspection was carried out regarding rust formation in the strand or change in the appearance of the coating. Two sections were cut after 10 months. The filler was removed from the strands, the wires cleaned and inspection via stereo microscope was performed.

2.3.2 Filler-coated Single Wires

Single wires with a length of 10 centimeters were cut from one of the seven-wire ~4 foot long steel strands. Samples of wires non-coated and coated with a thin layer (approx. 1 millimeter) of each of the filler were exposed to four different environmental conditions at Sea Tech. (1) The same metallic shelf located outdoors at the partially sheltered location; (2) an enclosed container located outdoors, with water at the bottom; (3) The Marine and Corrosion Lab at 22°C and high moisture (the wires were placed in plastic bins containing water and a plastic mesh); and (4) An elevated temperature room set at 32°C and high moisture (the wires were placed in plastic bins containing water and a plastic mesh). For each environmental condition, a total of 6 wires were prepared: 5 wires were coated with each of the 5 different fillers, and the last one was not coated (blank). These tests were performed in order to check the corrosion prevention effects of the

different fillers and see how corrosion evolved. Continuous visual inspection regarding rust formation in the wire or change in the appearance of the coating was performed. After at least 10 months of exposure, the wires were cleaned and a visual inspection performed (including stereo microscope observation).

The filler was mechanically applied to each wire segment. Each wire segment was then passed thru a transparent hose of 6 millimeter internal diameter. This process was repeated at least three times to ensure good filler coverage.

2.3.3 Filler-coated Single Wire Inoculated with Fungi

Single wires obtained by cutting 10 to 16 centimeter long pieces from the 4 foot steel strands, were coated with each of the fillers and sprayed with four different suspensions: a suspension with each fungi (*Fusarium oxysporum*, *Penicillium chrysogenum*, and *Aspergillus flavus*), and the other suspension was prepared with a mix of the three fungi. The wires were coated with filler as described in the above section. Once the wires have been coated and inoculated, the samples ends were covered with a piece of Parafilm tape and the ends secured with alligator clips. 1.5 milliliter distilled water was added to the wire coated in the hose (some contained the fungi suspension). A total of 50 samples were prepared including two sets of 25 samples (4 suspensions × 5 fillers + 1 control (water only) per filler). One set was exposed at the Marine and Corrosion Lab at 22°C and the other set was placed in an elevated temperature room set at 32°C. Both sets of samples were allowed to stand undisturbed. Exposure started on December 10, 2016.

Excess solution was observed during an inspection performed in January 2017. The excess solution inside the transparent tubing was removed and stored in small vials. The Nontribos coated samples (i.e., those sprayed with 4 suspensions and one with water) and all specimens coated with the other flexible filler and sprayed with *Fusarium oxysporum* were selected to be opened. This took place on January 25, 2017 (44 days after exposure started). The remaining layer of filler was removed and stored for future analysis. The coated wire specimens were then cleaned first with mineral spirits and then with an acid bath per ASTM G01. The rest of the wire samples were removed on May 31, 2017 (170 days of exposure) from the transparent tubing, cleaned with mineral spirits, and then to an acid cleaning. Pictures were taken including with a stereo microscope.

A second batch of samples were prepared in which the wires were coated differently. A small amount of each filler was placed in a glass container and the temperature of the filler increased using a hot-plate. Upon the filler becoming liquid the wire was dipped and allowed to slightly cool before removing. The coated wires were then placed in a transparent hose segment. The coated wires were then inoculated with the solutions described above, but a 0.6milliliter solution was sprayed into each sample. The exposure for these samples started on February 15, 2017 and were cleaned on July 18, 2017, thus the exposure lasted for 153 days.

2.3.4 Filler-injected Samples with Two or Three Single Wire Later Inoculated with Fungi

Single wires with a length of 6 inches were cut from a seven-wire steel strand segment. Polycarbonate tube segments were used with an internal diameter of 0.75 inches and an outside

diameter of 1 inch. Two or three wires were used per sample. Samples with wires were injected with each of the fillers, using a smaller funnel to direct the filler into the polycarbonate tube. Five samples per filler were prepared. One sample was sprayed with water and the other four with suspensions containing fungi (see description below). A total of 25 samples were prepared.

- Sanchem sprayed with water, F.O., A.F., P.C., and fungi mix
- Trenton sprayed with water, F.O., A.F., P.C., and fungi mix
- Nontribos sprayed with water, F.O., A.F., P.C., and fungi mix
- Civetea sprayed with water, F.O., A.F., P.C., and fungi mix
- Sonneborn sprayed with water, F.O., A.F., P.C., and fungi mix

The samples were exposed to laboratory humidity and temperature (approximately 65% RH and 22°C). The samples were sprayed 4 times on one side (top side). During March 2018, one wire was removed and cleaned for analysis after 200 days.

2.4 Electrochemical Tests

An electrochemical cell with one copper electrode, one magnesium electrode and one strand electrode (seven wires) was designed to assess the corrosion protection behavior of each of the flexible fillers. Figure 4 shows the cell which was made of clear plastic plates. First, the seven-wire strand was cut using a low speed diamond saw and the strand pieced cast in epoxy resin. After curing for 24 hours, the cross-section of the seven-wire strand were abraded using silicon carbide paper. A copper wire was connected to the each of the strand wires using a silver filled epoxy adhesive with low electrical resistivity applied and later covered with marine epoxy. As for the copper and magnesium electrodes, a hole was drilled and taped. Then a small 2-56 screw was placed for electrical connection.

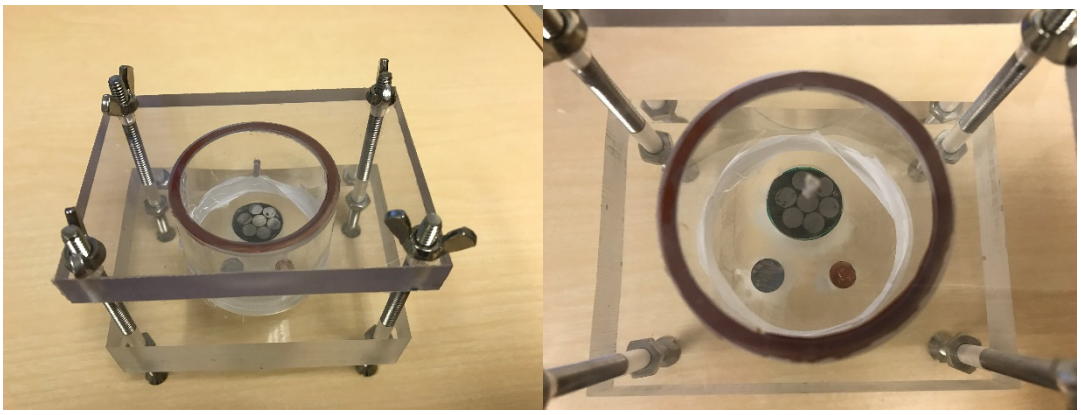


Figure 4. Electrochemical cell setup.

Two types of tests were conducted including impedance and galvanic current. A potentiostat was used to perform the tests.

To check if this set-up worked, tap water with a little bit of salt was be used as test solution. Once it was verified that the cell worked (i.e., there were no leaks and good measurements obtained), the cell was rinsed with distilled water. The seven-wire steel electrode and the other two electrodes

were then coated with the different flexible fillers and a simulated soil solution of pH 4.4 was added as test solution.

2.5 Larger Samples

This section describes the procedure used to inject samples that were prepared by heating the filler and then using a manual pump to inject the flexible fillers. The prepared samples were 4 feet long or 2 feet long samples. Samples were injected with each of the five flexible fillers. Most samples have 2 or 3 seven-wire steel strands.

A wood stand was used to hold the tubing. The stand was made by SRC-FDOT staff and it is shown in Figure 5. A similar stand was later prepared at FAU-SeadTech. The stand was made of 2 by 4 wood pieces and painted blue. The height of the stand is about 4 feet and it has an incline that allows it to hold the 4 foot long tubing. Two types of tubing were used to prepare the samples: transparent polycarbonate tubing and HDPE pipes. The internal diameter of these pipes is 2 inches. To have a good sealing at each end, two type of caps were used. For HDPE pipes, white PVC caps were used. PVC glue was used to secure the white PVC-cap to the HDPE duct. Whereas for the polycarbonate tubing, black rubber-caps were used. The clamp surrounding the cap was adjusted to provide a good seal. Two lengths were cut and injected with the flexible filler for polycarbonate tubing segments. Some polycarbonate tubes were 4 feet long, and the other polycarbonate tube segments were cut to 2 feet long. The HDPE segments were a little longer than 4 feet long. Once the tubing was placed in the wood stand, steel strands were inserted into the pipes. Two strands (in a few cases three strands) were inserted for each pipe sample. The strand segments were 2 feet or 4 feet long depending on the tube length. Figure 6 shows the tubing materials and some of the seven-wire steel strand segments used. The flexible fillers were heated and injected to each sample.

Figure 5. Left: Stand used with tubing ready to be injected; Right: Caps used.

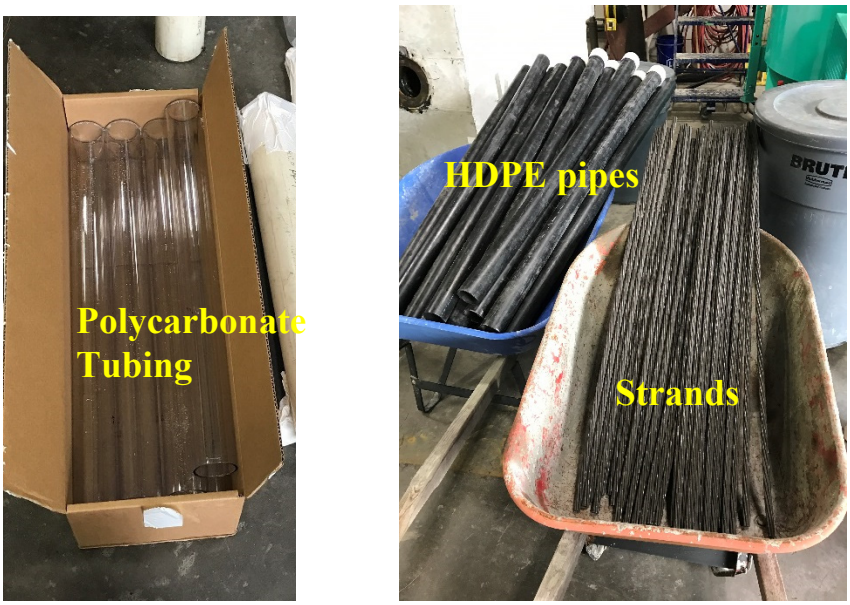


Figure 6. Polycarbonate tubing, HDPE tubing, and steel strands used

2.5.1 Heating

To heat each of the flexible fillers, a 5 gallon (or 10 gallon) metal bucket was used. First, the solid grease was transferred into the metal bucket, then heated with an electrical heater wrapped around the bucket as shown in Figure 7. Dependent on the flexible filler type, the heating duration ranged from 6 to 12 hours.

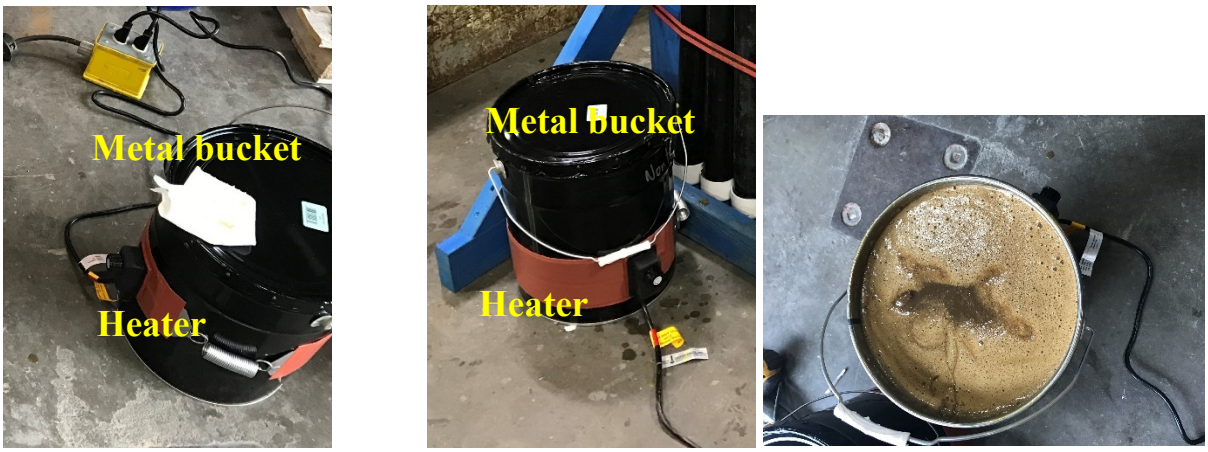


Figure 7. Heater and metal bucket.

2.5.2 Injection

The injection was made using a manual pump as shown in Figure 8. During the wax injection, the strands were separated to make good contact of wax with each strand. The wax was injected to a level that completely covered the strands in each tube. The manual pump was cleaned before each injection using mineral spirits (paint thinner).

Figure 8. Flexible filler injection and a picture of the pump used.

2.5.3 Cooling Down and Capping

After injection, the pipes were kept open to cool down the smelting fillers for about 2 hours before placing the top cap. Figure 9 shows on the left some samples while waiting for cooling down and on the right a picture after the samples have cooled down with both caps. Figure 10 shows a top view after completing filler injection, prior to placing the top cap.

Figure 9. Left: Cooling down of 4-foot samples; Right: After top cap had been placed.

Figure 10. Top view of flexible filler cooling down prior to capping.

The samples prepared at FDOT-SRC were transported to FAU-Sea Tech. Some of the samples were actually prepared at FAU. Holders/supports to simulate relevant angles during exposure were prepared. Concrete blocks were used in one case, and an aluminum I beam in the other case (at the semi-sheltered location). Two types of end-covers (caps) were used. One end-cover was made of PVC, and the other end-cover was made out of rubber with an O-ring clamp. Most of the caps were removable. Moisture was periodically sprayed on selected samples.

The paragraphs that follow list the samples by exposure location grouped per sample type. Each subsection that follow contains pictures of the exposure setup and describes the type of sample, whether the sample was sprayed, what type of solution was used, and how much was used initially. It also indicates if additional wetting took place and with what solution. The results section presents pictures with the caps removed from both sides. On each sample, only one side was sprayed with solution. Figure 11, Figure 12, and Figure 13 show pictures of the samples placed outdoors under the stairs on the west side of Sea Tech. It can be observed that the tubing is made of high density polyethylene. There are samples that are 4 inches in diameter and others that are 2 inches in diameter.

Figure 11. Four-inch and 2-inch diameter samples injected with flexible filler.

Figure 12. Closer view of the 4-inch diameter samples injected with Trenton at FDOT Structures lab

Figure 13. Two-inch diameter HDPE samples injected with various fillers and preconditioning.

1) Location: Outdoors west side of Sea Tech under the stairs (4 inch diameter samples all injected with Trenton and 2 inch diameter samples injected with the different fillers are exposed here)

a) *4 inch diameter samples injected with Trenton*

Figure 12 shows a closer view of the 4 inch diameter samples. The segments provided by FDOT-SRC were between 8 and 9 feet long, and an additional cut was made to have manageably sized specimens (approx. 4 feet long each). Six of these samples were sprayed and four were not sprayed. Table 11 shows that one sample was sprayed with water, two samples were sprayed with a fungi mix, and three samples with each of the three fungi. Approximately 0.6 milliliter of solution was sprayed on May 9, 2017. This was the date of the initial spray. Additional solution spray took place about every three weeks. The two samples sprayed with a fungi mix, continued to be sprayed with this type of solution and the other four samples were sprayed with water on subsequent inspections. Only one side of the sample was sprayed. The sprayed surface was the elevated end of each specimen.

Table 11. Four-inch diameter samples injected with Trenton

	fungi mix	H2O	Fungi 1 (F.O.)	Fungi 2 (A.F.)	Fungi 3 (P.C.)	Blank
Quantity	2 Sprayed with ≈ 0.6 ml of solution	1 Sprayed with ≈ 0.6 ml of solution	1 Sprayed with ≈ 0.6 ml of solution	1 Sprayed with ≈ 0.6 ml of solution	1 Sprayed with ≈ 0.6 ml of solution	4 Not sprayed
Start Date	05/09/2017 Sprayed with fungi mix every 3 weeks	05/09/2017 Sprayed with water every 3 weeks	05/09/2017 Sprayed with water every 3 weeks	05/09/2017 Sprayed with water every 3 weeks	05/09/2017 Sprayed with water every 3 weeks	05/09/2017 Not sprayed

b) *Samples with flexible fillers injected in 2 inch diameter HDPE tubing*

Table 12 lists the number of samples injected with each type of filler and what type of solution that was used. The note below the table indicates the meaning of NS and RS. Samples marked in the as injected with fungi were injected with the fungi mix the first time and also for subsequent sprays.

Table 12. Two-inch Diameter Samples Injected with Various Flexible Fillers

Sample/Filler	Sanchem (NO-OX-ID-NG)	Sonneborn (Visconorust-2090-P)	Gähringer (Nontribos VZ Inject)	Civetea (Cirinject CP)	Trenton
N.S.	1 Fungi	1 Fungi	1 Fungi 1 Water	1 Fungi 1 Water	1 Fungi 1 Water
R.S.	1 Water	1 Water	1 Water	1 Water	1 Water
Over heat (No strands)	-	-	-	1 Fungi 1 Water	1 Fungi 1 Water
Start Date	05/09/2017 Sprayed with solution every 3 weeks (≈ 0.4milliliter)	05/09/2017 Sprayed with solution every 3 weeks (≈ 0.4milliliter)	05/09/2017 Sprayed with solution every 3 weeks (≈ 0.4milliliter)	05/09/2017 Sprayed with solution every 3 weeks (≈ 0.4milliliter)	05/09/2017 Sprayed with solution every 3 weeks (≈ 0.4milliliter)

Note: Rusted Strand [RS]: Strands were exposed outdoors at Sea Tech for 18 days until they were homogeneously rusted. Neat Strand [NS]: strand without any pre-conditioning. (NC also indicates that no conditioning was done on the filler). Two samples were injected with overheated flexible filler (Civetea and Trenton only) and one sample was sprayed with fungi. The other sample was sprayed with water.

Figure 14. Two-inch diameter samples injected with various flexible fillers.

2) Samples exposed upstairs at the terrace/patio semi-sheltered location: a) Samples with flexible filler injected in polycarbonate 4 footfoot tube. b) Single coated strand samples that are in PVC pipes.

a) *Samples with flexible filler injected in polycarbonate 4 foot tube.* One sample was prepared with strands conditioned (i.e., strands exposed outdoors during 2016) and two samples were prepared with "as received" strands (i.e., with no conditioning). Two or three strand segments were introduced in polycarbonate tubing and then injected with the corresponding filler. Once the samples were placed at the semi-sheltered location, selected samples were sprayed with the solution indicated in Table 13. Only one side of the sample were sprayed. Table 13 lists the type of spray that was used. Fungi mix solution was used to spray the appropriate samples every two weeks.

Table 13. Two-inch Diameter Samples – Polycarbonate tube

Sample/ Filler	Sanchem (NO-OX-ID-NG)	Sonneborn (Visconorust-2090-P)	Gähringer (Nontribos VZ Inject)	Civetea (Cirinject CP)	Trenton
Sprayed with fungi mix NC (≈ **0.4 ml**)	1	1	1	1	1
Sprayed with Water NC (≈ **0.4 ml**)	1	1	1	1	1
C1 sample Not sprayed	1	1	1	1	1
Start Date	04/07/2017 Sprayed every 3 weeks	04/07/2017 Sprayed every 3 weeks	04/07/2017 Sprayed every 3 weeks	04/07/2017 Sprayed every 3 weeks	04/07/2017 Sprayed every 3 weeks

Table 13 lists the samples that were sprayed either with fungi mix or water. Figure 14 shows that two of the three samples per filler type were supported by an aluminum I-beam. The third sample per set was placed on a shelf. Figure 15 shows three samples exposed at the partially sheltered site located upstairs (terrace) that were injected with Trenton. The sample from the shelf was momentarily moved next to the other two samples injected with Trenton.

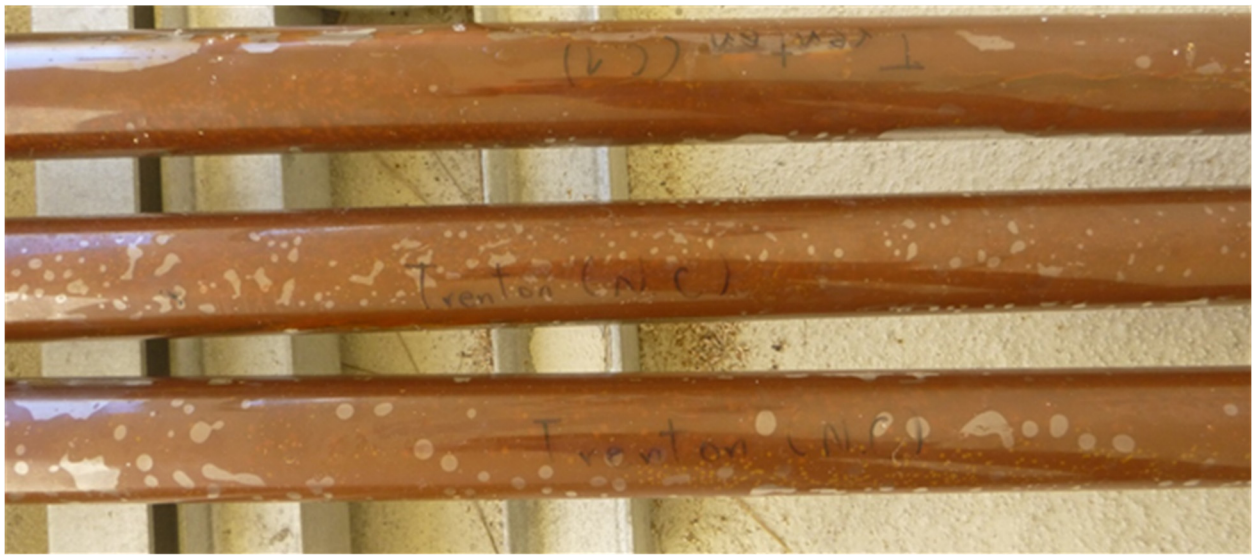


Figure 15. Two-inch diameter 4-foot long samples injected with Trenton (two NC and one C1 conditions)

b) *Single coated strand samples that are in PVC pipes.*
The single coated strands were provided by SRC. The coated strands with Civetea and Nontribos were part of the earlier mock- set-up described earlier. The strands coated with Trenton were provided by FDOT-SRC as tendon segments with 19 strands in a 4 inch diameter HDPE duct. These samples are part of a more recent mock-up set-up. The samples were exposed at the semi – sheltered location on April 3, 2017. The solution was first applied on May 5, 2017. Table 14 lists the number of samples

Table 14. Single Coated Strand Exposed Inside a PVC pipe

Sample/ Filler	Civetea (Cirinject CP)	Gähringer (Nontribos VZ Inject)	Trenton
Sprayed with fungi mix (≈ **0.6 ml**)	1	1	1
Sprayed with Water (≈ **0.6 ml**)	1	1	1
Non sprayed "Blank"	-	1	1
Start Date	05/05/2017 Sprayed with water every 3 weeks	05/05/2017 Sprayed with water every 3 weeks	05/05/2017 Sprayed with water every 3 weeks

After inoculation, the strands were introduced in a PVC pipe with 1.75 centimeter diameter). Each PVC pipe has PVC caps on each end that prevent additional moisture from entering. One strand coated with Trenton, one with Civetea, and one with Nontribos were inoculated with the fungi mix. The fungi mix was applied by using a spray bottle. The suspension was sprayed around the entire surface of the coated strand (12 times for a volume of 1.5 milliliter). One coated strand per filler type was sprayed with water, by spraying 1.5 milliliter of distilled water on the surface area of each coated strand. Additional wetting took place about every three weeks. Table 14 lists the

samples initial spray date and how often the samples were wetted. For this set of samples: The strands previously sprayed are periodically removed from the PVC and the entire surface is sprayed. The blank samples were not wetted, but they were removed for periodic inspection on the days that the other samples were visually inspected. Visual inspections took place while the strands were out, and in a few instances the surface condition of the strand was documented with pictures (see Results section). Samples selected for forensic examination were exposed for approximately 280 days. Sections 10 to 12 centimeter long were cut off from each of the coated strands sprayed with fungi. Sections were also cut off from samples sprayed with water and coated with Civetea and Nontribos, but not from the Trenton coated strand. The filler was manually removed and then mineral spirits were used to remove any remaining filler. The wires showing the most corrosion were selected for chemical cleaning using ASTM G1-03.

3) Location: Indoor Laboratory Exposure: 2-foot-long Samples.
One sample per filler type was prepared with contaminated filler. This means a certain amount of these fillers were exposed outdoors at the semi-shelter location and particulates were able to reach the top of the filler. This outdoor exposure took place during early 2016. Some of the samples had pre-rusted steel strands referred to as RS (rusted strands). Table 15 describes the number of samples injected into a 2-foot-long polycarbonate tube. It also indicates what type of spray was used and how often this took place. Figure 16 and Figure 17 show the 2-inch diameter samples that are 2-foot-long and that were injected with various flexible fillers described in Table 15.

Table 15. Two-foot-long and 2-inch Diameter Samples Injected with Various Fillers Exposed in the lab.

	Sanchem (NO-OX-ID-NG)	Sonneborn (Visconorust-2090-P)	Gähringer (Nontribos VZ Inject)	Civetea (Cirinject CP)	Trenton
CF & NS (≈ **0.4 ml)**	1 water	1 water	1 water	1 water	1 water
NF & RS (≈ **0.4 ml)**	1 water	1 water	1 water	1 water	1 water
NF & NS (≈ **0.4 ml)**	1 fungi mix	1 fungi mix	1 fungi mix	1 fungi mix	1 fungi mix
Start Date	04/26/2017 Sprayed with water every 5 weeks	04/26/2017 Sprayed with water every 5 weeks	04/26/2017 Sprayed with water every 5 weeks	04/26/2017 Sprayed with water every 5 weeks	04/26/2017 Sprayed with water every 5 weeks

Contaminated filler (CF): Fillers were exposed for 120 days outdoors. The outdoor exposed fillers were collected, heated and injected instead of the neat filler (i.e., as received filler). Rusted Strand (RS): Strands where exposed outdoors at Sea Tech for 18 days during the rainy season until they were homogeneously rusted. Neat filler (NF): filler without any pre-conditioning. Neat Strand (NS): strand as received.

Figure 16. Two-inch diameter, 2-foot long samples stored in the laboratory

2.6 Forensic Examination Tests

2.6.1 Visual Inspection

Samples for which the filler was injected (i.e., 2 foot and 4 foot samples) were subjected to periodic visual inspections. The caps were removed and the surface condition documented. On selected instances pictures were taken after removing the caps (in some cases pictures were taken from both ends).

2.6.2 Chemical Cleaning of Wires after Exposure

Forensic examination of the coated strands/wires occurred upon exposure completion. A visual inspection including a microscopic analysis was performed after cleaning the strands/wires. The wires were subjected to a chemical cleaning bath. This procedure was done in order to remove the corrosion products. Before submerging the wires in the bath, the excess flexible filler was removed manually and then using mineral spirits. The cleaning solution proportion was 200 milliliter of DI water, 200 milliliter hydrochloric acid (HCl, sp gr 1.19) and 0.7 g of hexamenthylene tetramine, the cleaning procedure was carried out in the laboratory fume hood at 21°C.

2.6.3 SEM

Selected corrosion sites of wires from filler-coated strands and single wires of injected small polycarobonate samples were inspected using a scanning electron microscope. A small section about 2 centimeter long was cut off from the selected wire and then pictures were taken from 26x× to 1,000 x.

Figure 17. Two-inch diameter, 2 foot long samples stored in the laboratory at a shelf

3.1 Filler Deterioration Tests

3.1.1 Flexible Filler Exposed Outdoors

After 44 days of exposure within the covered container located outdoors, the filler Civetea started changing texture and color. After 64 days of exposure, it presented a completely different color (Figs. 18a-c). The filler sample was not removed. It remained under exposure with the other fillers. The smaller containers with filler Civetea exposed at the semi-sheltered area also changed its texture and color, but this was observed after 60 days of exposure (Fig. 18a-b).

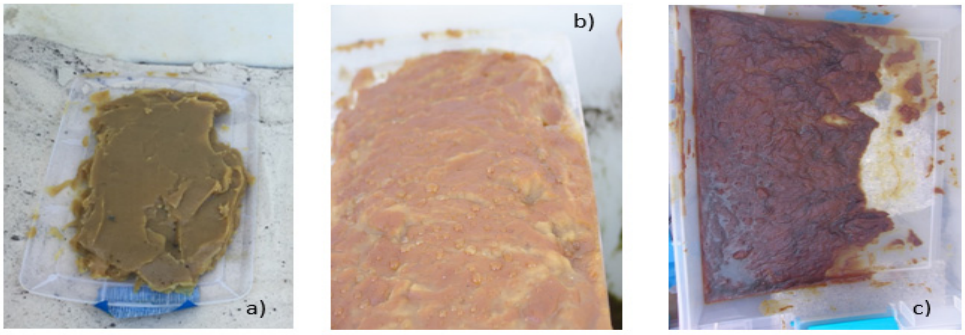


Figure 18. (a) Civetea original color; (b) After 44 days of exposure; (c) After 64 days of exposure.

Figure 19. (a) Civetea original color (April 11 2016); (b) Civetea after 60 days of exposure.

After approximately a year of exposure the color of the Civetea filler remains as the one shown in figure 19.b (i.e., darker than the original color). With respect to the other flexible fillers, none of the samples presented a change of texture or color as Civetea did. Some of the filler samples at the semi-sheltered location presented contamination by bugs, particulates, dust pieces, or palm fronds, even small amounts of water drops were observed (likely due to heavy rain and wind, or even dew) at the corners or spaces of the containers where the filler wasn't applied uniformly. The contaminated outdoor fillers were removed from exposure and used as injection material for selected samples. Some of the 2 foot long samples used these fillers.

3.1.2 Inoculated Fillers

None of the samples left in the elevated temperature (40°C) room presented visual presence of the fungi, i.e., they did not spread (June 2017). Nevertheless, the samples placed in the laboratory at 21°C showed presence of two out of three of the fungi species. The first visual sign of fungi was observed in the Petri dish with Trenton sprayed with *Aspergillus flavus*, after 25 days, followed by Civetea sprayed with *Aspergillus flavus* and Sonneborn sprayed with *Fusarium oxysporum* after 34 days of exposure. After 60 days of exposure, it was possible to observe that *Fusarium oxysporum* was able to propagate in Trenton (Figure 20), Sonneborn, and Nontribos (Figure 22). The species *Aspergillus flavus* was able to use the five fillers as carbon source in order to grow. Figure 20 and Figure 22 shows additional fillers sprayed with fungi as of April 2017. The samples were inspected during June/July of 2017 and remained the same, with no additional presence of fungi species in the microcrystalline waxes.

Figure 20. Left: Trenton with *Aspergillus flavus*; Middle: Trenton not inoculated; Right: Trenton with *Fusarium oxysporum* (April 2017)

Figure 21 and Figure 23 show the same samples, but the pictures were taken during July 10, 2018. Figure 24 shows a picture taken July 10, 2018, on Sonneborn with *Aspergilus flavus*. A visual inspection took place during July 2018, and it appears that fungi did not propagate any further. Moreover, in some cases, it appears that the region with fungi was covered by a smaller area compared to that observed in June 2017. Some of the images taken in 2018 used flash, and thus, the filler appear to be a lighter color.

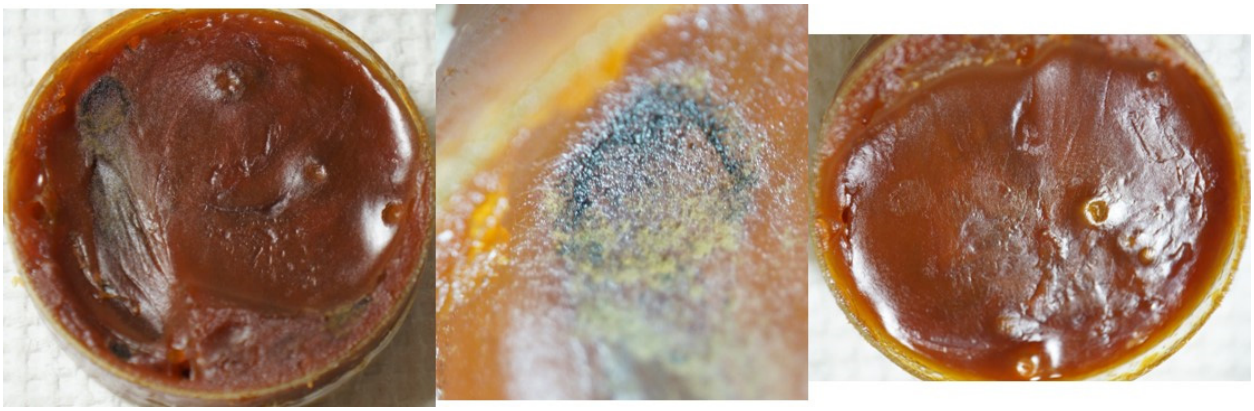


Figure 21 Left: Trenton with *Aspergillus flavus*; Middle: Trenton not inoculated; Right: Trenton with *Fusarium oxysporum* (July 2018)

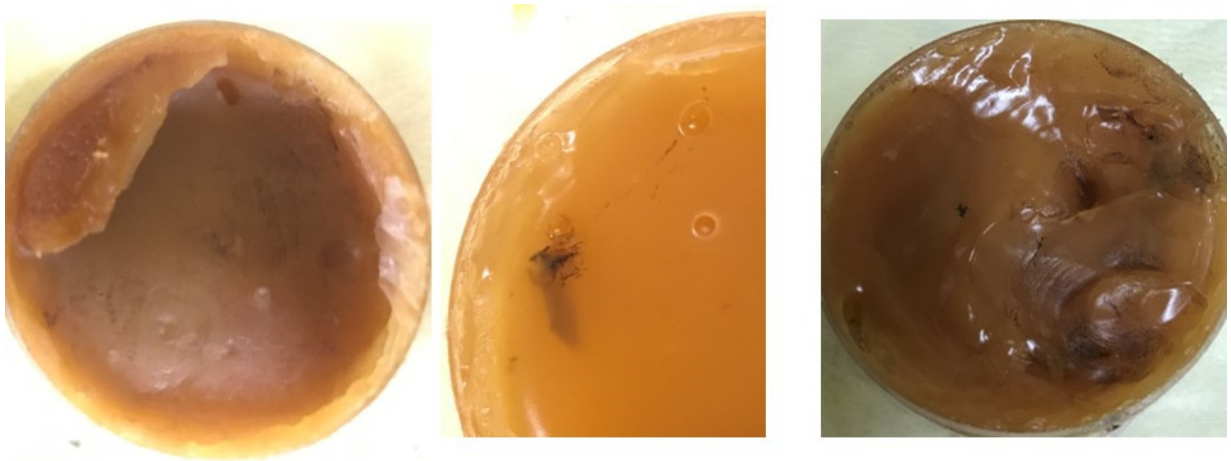


Figure 22 Left: Civetea with *Aspergillus flavus*; Middle: Nontribos with *Fusarium oxysporum*; right: Nontribos with *Aspergillus flavus* (April 2017)

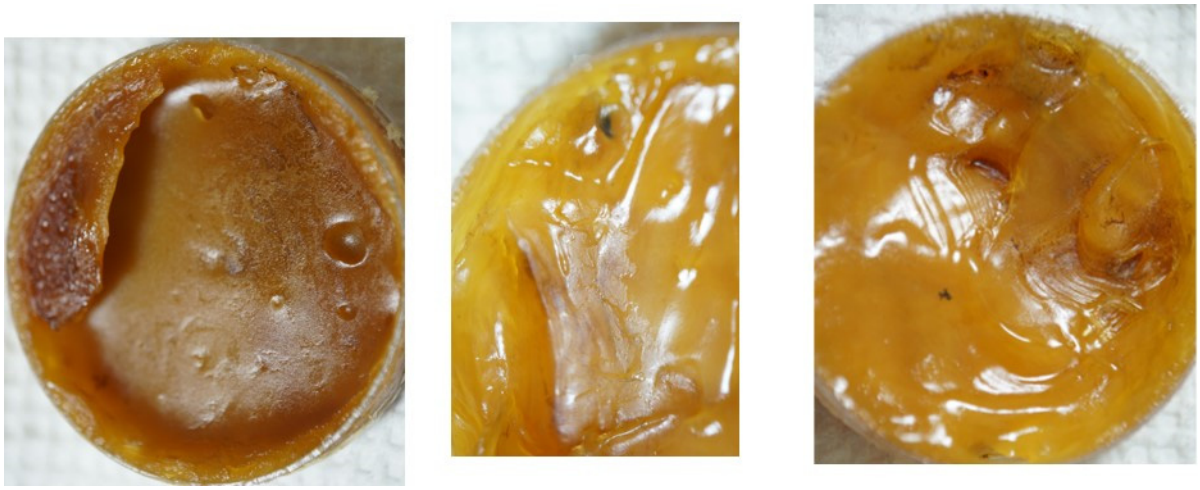


Figure 23 Left: Civetea with *Aspergillus flavus*; Middle: Nontribos with *Fusarium oxysporum*; right: Nontribos with *Aspergillus flavus* (July 2018)

Figure 24. Sonneborn with *Aspergillus flavus* (July 2018)

3.1.3 FTIR

Fourier transform infrared spectroscopy (FTIR) of each of the microcrystalline waxes has been obtained (neat condition only) (Figure 25 and Figure 26).

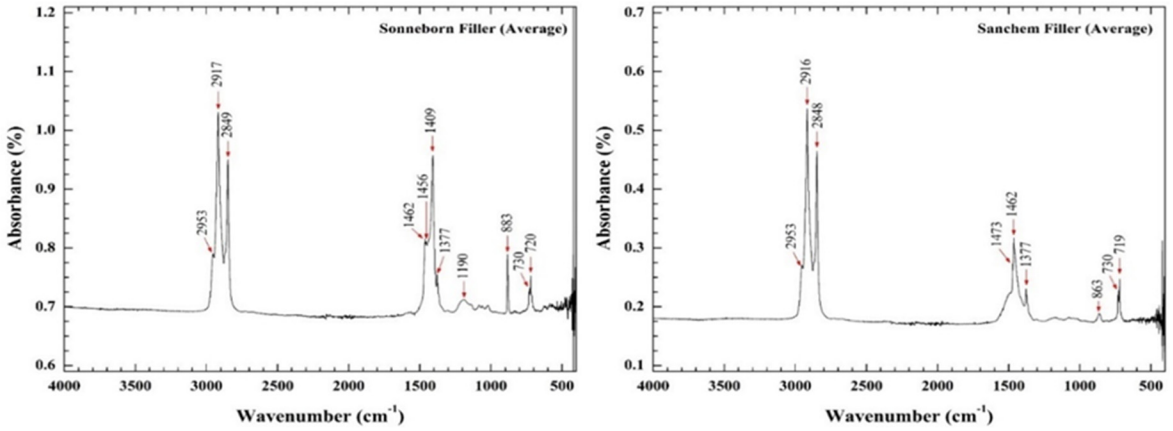


Figure 25. FTIR neat: Sonneborn and Sanchem

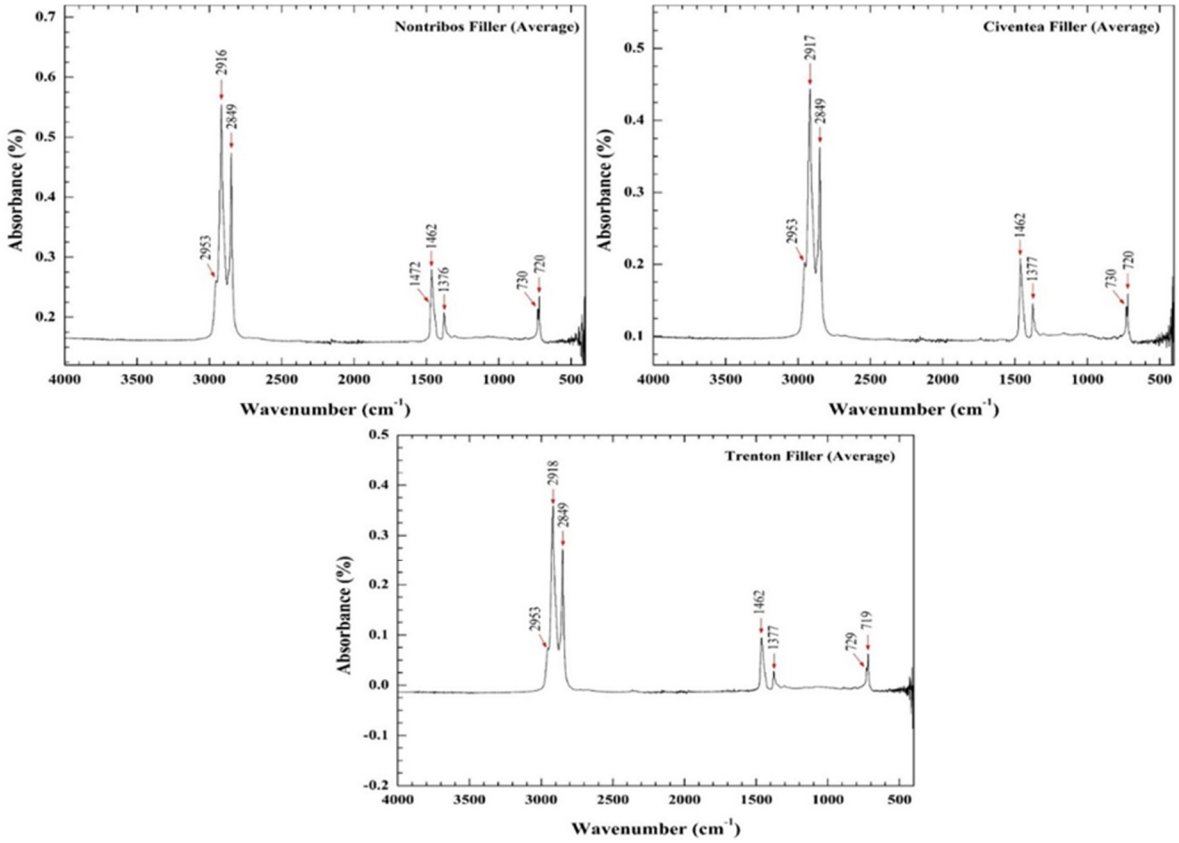


Figure 26. FTIR neat: Nontribos, Civetea, and Trenton

Table 16. Peaks observed in the FTIR scans of the neat fillers.

Microcrystalline wax	# of peaks	2800 to 3000 cm^{-1}	1100 to 1500 cm^{-1}	700-800 cm^{-1}
Civetea	7	2953 2917 2948	1462 1377	730 720
Nontribos	8	2953 2916 2849	1472 1462 1376	730 720
Sonneborn	11	2953 2917 2849	1462 1456 1409 1377 1190	883 730 720
Sanchem	9	2953 2916 2848	1473 1468 1377	863 730 719
Trenton	7	2953 2918 2849	1462 1377	729 719

Table 16 lists the number of peaks and the wave number location at which the peaks were observed on the different microcrystalline waxes. All fillers had three peaks between 2,800 and 3,000 (cm^{-1}), the number of peaks at the other ranges varied depending on the filler.

3.2 Exposure Tests of Coated Strand and Wires

3.2.1 Filler-coated Strands Exposed Outdoors

Strand samples coated with Nontribos and Civetea segments were exposed on April 11, 2016 at both outdoor environmental conditions. None of the samples in the enclosed container presented rust by day 353 (when these strands were moved to the partially sheltered area or used to be inoculated and placed inside a PVC tube). With respect to the samples exposed outside at the semi-sheltered location, the strand coated with Nontribos started presenting rust spots on April 25, 2016 (14 days after exposure started). See Figure 27 that shows the rust spots starting to appear at the corners. By May 13, 2016 (34 days of exposure), rust spots were present in other areas of the strand as well (see Figure 28).

Figure 27. Samples coated with Nontribos, April 25, 2016

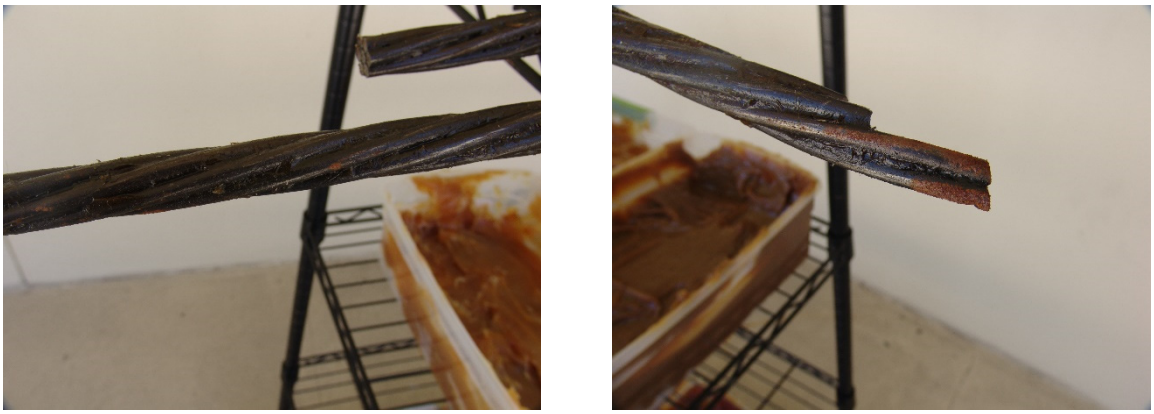


Figure 28. Samples coated with Nontribos, May 13, 2016

By April 18, 2017 it was possible to appreciate that rust had covered a large part of the surface of the strands (see Figure 29) coated with Nontribos. Figure 30 shows how the strands look by the end of June 2017. Figure 31 shows a couple of pictures of the strands coated with Nontribos taken in July 2018. Corrosion propagated and it appears that some wires suffered cross-section loss. Figure 32 shows a close-up picture for a section that showed corrosion. This picture is brighter because it was taken with a macro lens and flash.

Figure 29. Samples coated with Nontribos, April 18, 2017

Figure 30. Samples coated with Nontribos, June 26, 2017

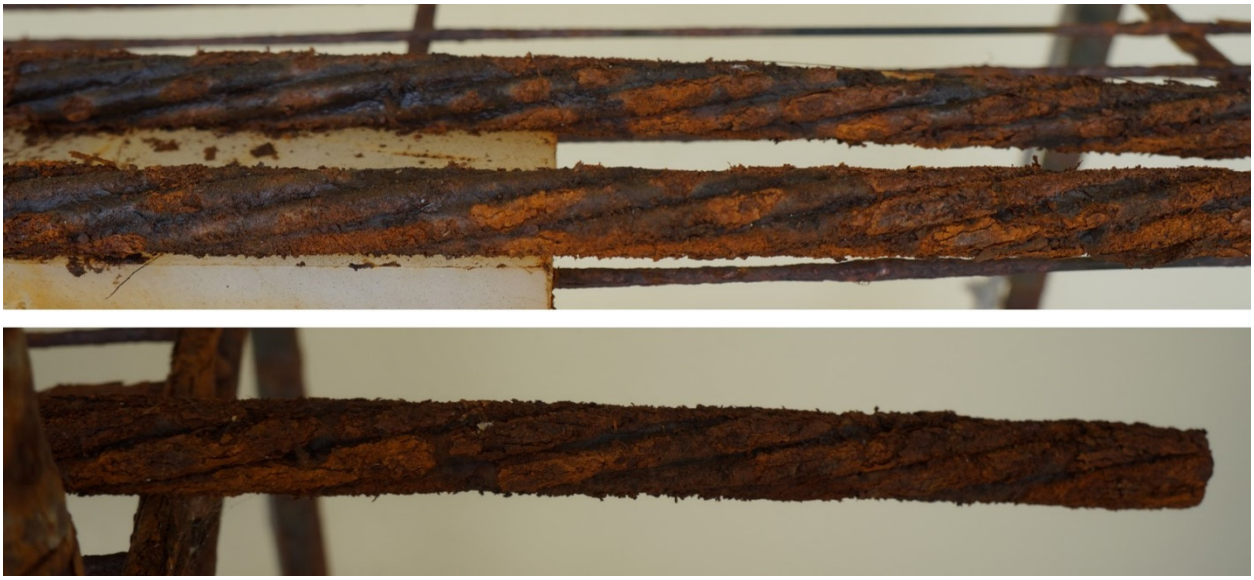


Figure 31. Samples coated with Nontribos, July 10, 2018

Figure 32. Samples coated with Nontribos, July 10, 2018 (close-up)

Strands coated with Civetea at the semi-sheltered location started presenting rust spots on July 12, 2016 (92 days after exposure started). This can be seen in Figure 33. The picture on the right shows the surface condition in November 2016.

Figure 33. Samples coated with Civetea. Left: July 12, 2016; Right: Nov 4, 2016.

After exposure for a year, rust is visible in some areas but there are other areas with a layer of the filler as well (Figure 34) as no signs of corrosion spots. Figure 35 shows how the strands look by the end of June 2017. Figure 36 shows the surface condition at two different spots with images taken during July 2018. It appears that the sample on the back (closest to the wall) has developed more corrosion spots than other coated strand. Still, there are several spots with corrosion on it. It is possible that the filler layer was thicker on the sample with fewer corrosion spots. Figure 37 shows a close-up for each of the segments, the pictures were taken with the flash on.

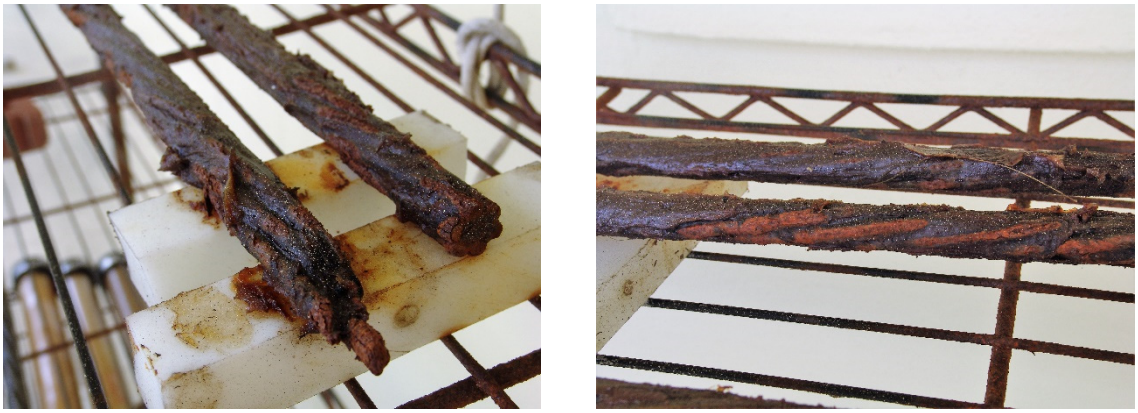


Figure 34. Samples coated with Civetea, April 18, 2017.

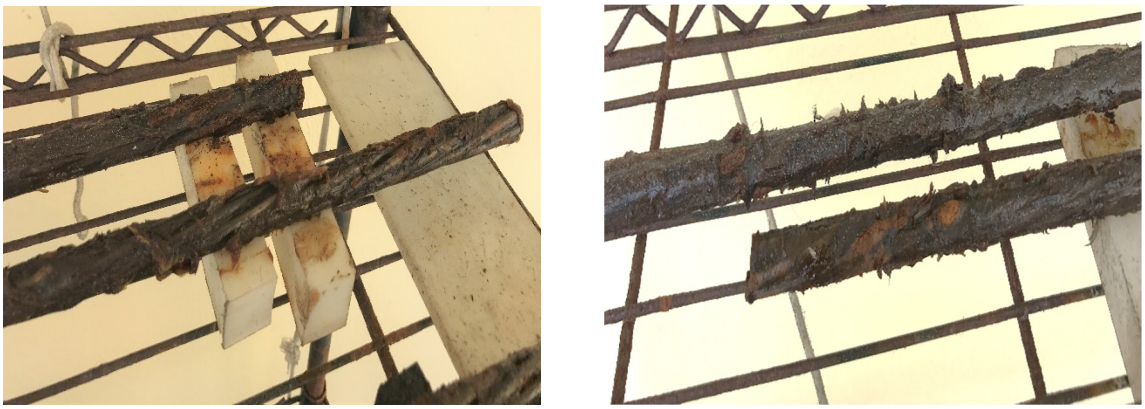


Figure 35. Samples coated with Civetea, June 26, 2017.

Figure 36. Samples coated with Civetea, July 10, 2018

Figure 37. Samples coated with Civetea, close-ups, July 10, 2018

Strands coated with Trenton were exposed at both sites on December 12, 2016. The strands exposed in the enclosed container did not show corrosion spots. These strands were then moved to the semi-sheltered area during early April 2017. Nevertheless, the filler-coated strand specimens exposed at the semi-sheltered location started presenting signs of corrosion on April 11, 2017 (120 days after initial exposure) (Figure 38). Figure 39 shows pictures taken during July 11 2017 and illustrate the condition of the coated strand at that time and that additional corrosion spots are visible: Figure 40 shows the state of the strands on July 10, 2018 and Figure 41 shows a close-up of the coated segment on the front.

Figure 38. Samples coated with Trenton, April 18, 2017

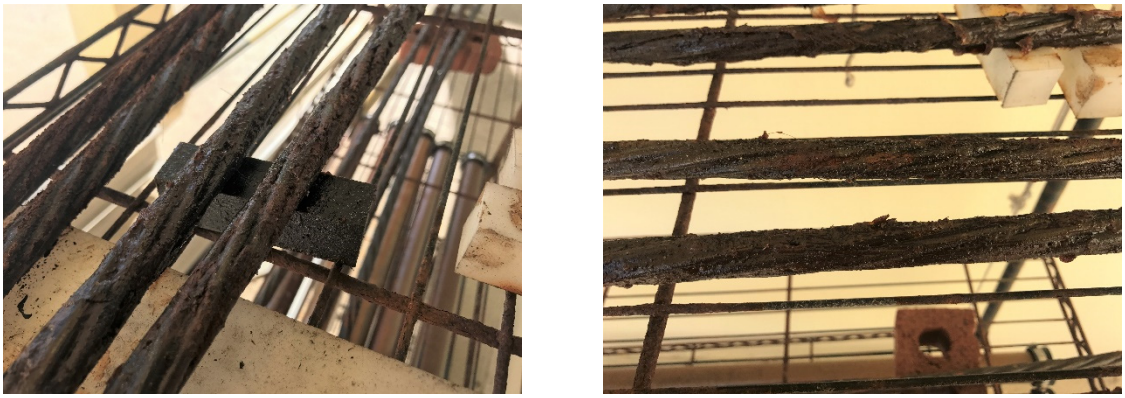


Figure 39. Samples coated with Trenton, July 11, 2017

Figure 40. Samples coated with Trenton, July 10, 2018

Figure 41 Close-up of sample coated with Trenton, July 10, 2018

After exposure for 389 days, two 12 centimeter long pieces at one of the edges of the strand and the following segment were cut off from one of the strands coated with Civetea and with Nontribos. Once the pieces were cut off, the 12 centimeter long specimens were cleaned with mineral spirits, in order to remove the filler or what was left of the filler after the exposure. An acidic cleaning bath was prepared and used to remove corrosion products. In Figure 42, it is possible to appreciate that even when rust was present on the outer wires of the strand, there is not much flexible filler remaining. Figure 42.(c) shows that the central wire was still covered with the filler. This was the pattern expected but it was only seen in the specimen coated with Civetea. Upon cleaning the wire strands coated with Civetea, the inner side of the wires showed that in some cases corrosion had initiated.

Figure 42. (a) Specimen coated with Civetea (12 centimeter) outside view; (b) Inner view; (c) Central wire.

For the specimens coated with Trenton, two 13 centimeter long pieces were cut off after 182 days of exposure. As expected, the inner side of the wires and the central wire still had some of the flexible filler (Figure 43).

Figure 43. Strand coated with Trenton (182 days after Exposure).

For the wire specimens coated with Civetea and Trenton, the central wire presented some corrosion. It's possible that in the process of the injection the filler didn't go through the whole inner part of the strand or the layer in the corner of the strand was thin letting particles enter to the location of the center wire from that point. In Figure 43, it can be observed that even though the central wire still has some of the flexible filler covering it, at some locations the central wire presented some corrosion. After cleaning the wire segments, all wire samples were observed via a stereo microscope. The following section shows the central wire and two of the other wires. Appendix A shows a collage of the observations on additional cleaned wires that were exposed at the semi-sheltered location.

Specimens coated with Nontribos showed that corrosion covered aproximately 40% of the wire surface of the external wires and very low corrosion extent on the central wire (less than 5%) for both the edge segment (Figure 44) and the following segment (Figure 45).

Figure 44. Wires from the strand coated with Nontribos, (a) Wire 6; (b) Central wire; (c) Wire 4.

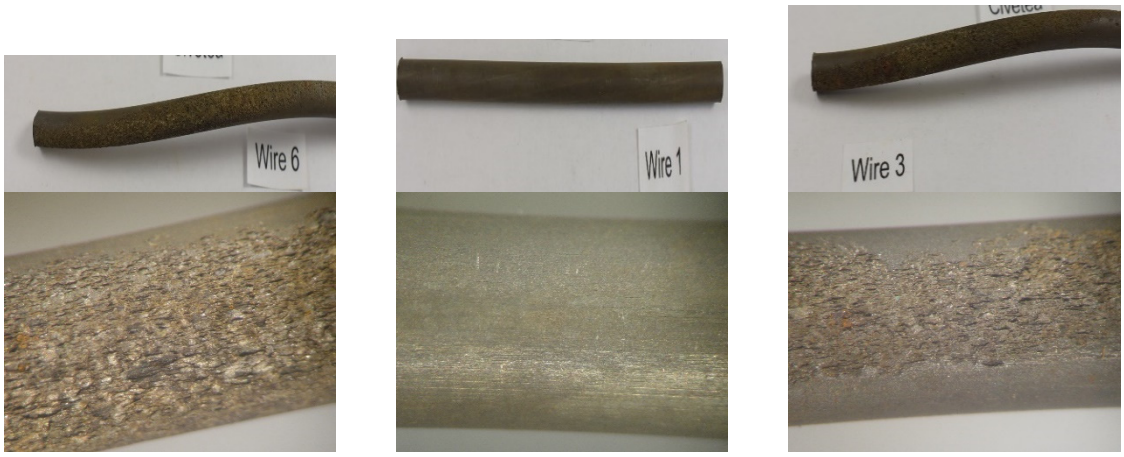


Figure 45. Wires from the strand coated with Nontribos, (a) Wire 6; (b) Central wire; (c) Wire 3.

Specimens coated with Trenton presented evidence that aproximatly 20% of the area corroded on the external wires and it showed corrosion coverage in around 10 % of the area for the central wire for both the corner segment and the following segment. Strands coated with this filler were exposed for a shorter period of time. (See Figures 46 and Figure 47). The depths of the corroding sites were significantly shallower than what was observed for wires coated with Nontribos.

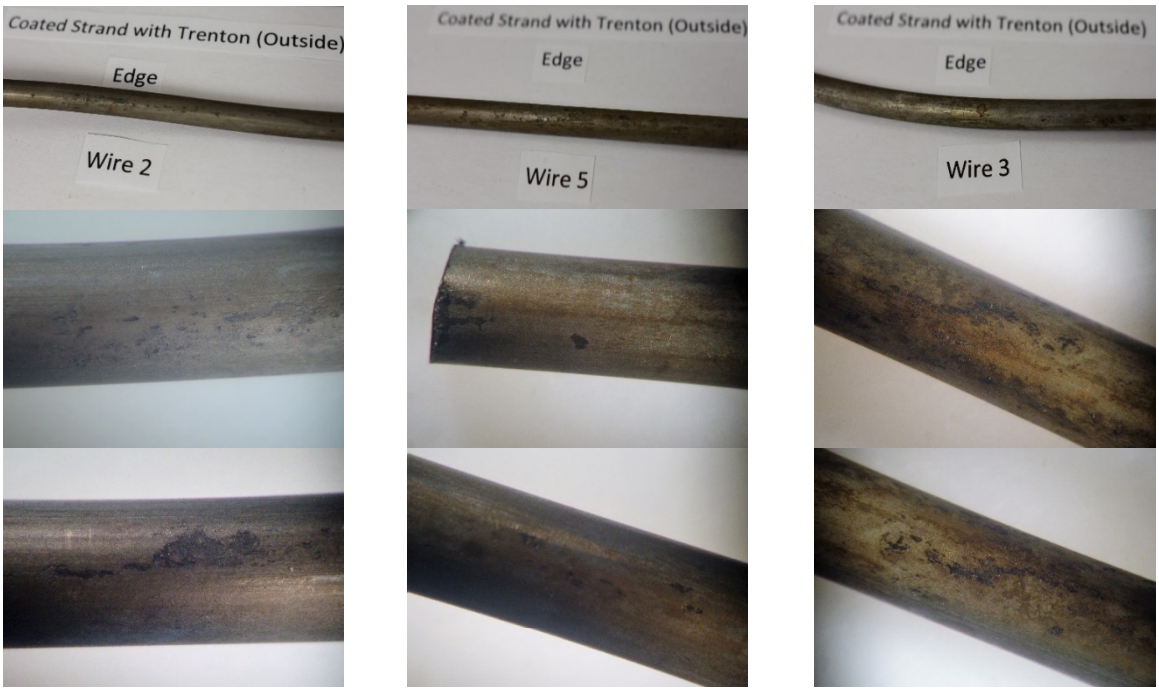


Figure 46. Wires from the strand coated with Trenton (edge): (a) Wire 2; (b) Central wire; (c) Wire 3.

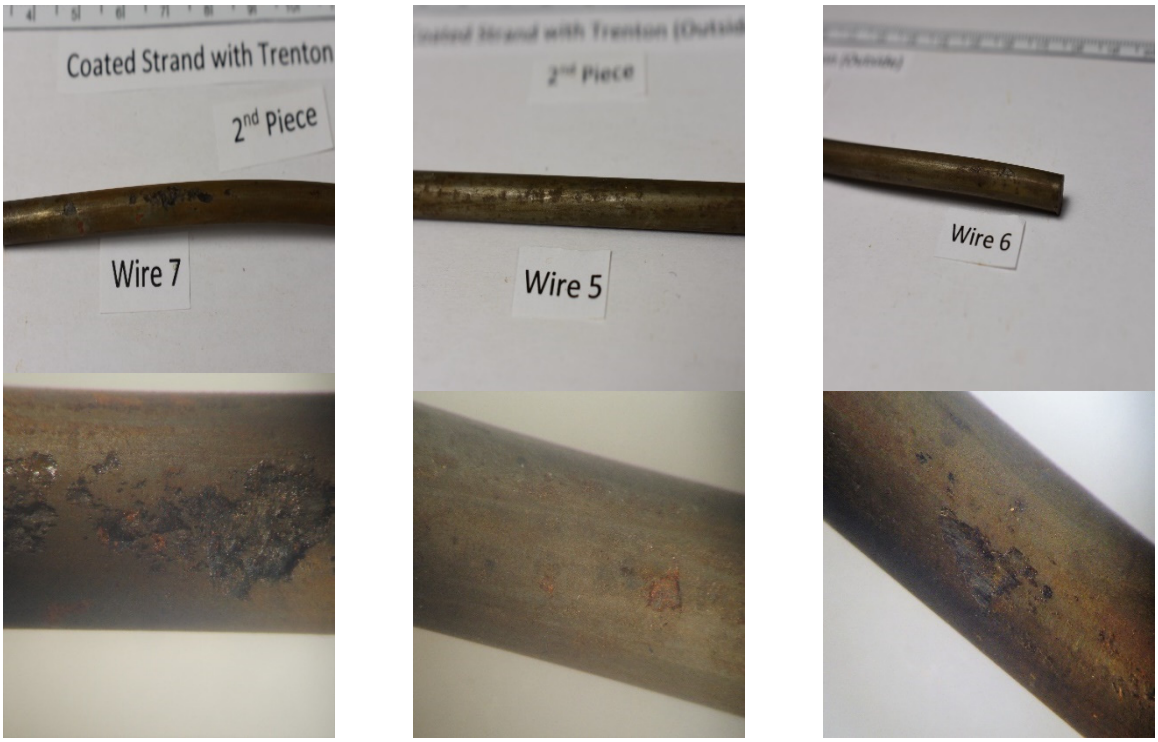


Figure 47. Wires from the strand coated with Trenton: (a) Wire 7; (b) Central wire; (c) Wire 3.

For the specimen coated with Civetea, the following figures (Figure 48 and Figure 49) show that the central wire presented significantly more rust than that observed on specimens coated with Sanchem or Trenton. Specimens coated with Civetea presented corrosion on aproximatly 30% of the area on the external wires. The central wires showed corrosion on approx. 10% of the area

Figure 48. Wires from the strand coated with Civetea (edge): (a) Wire 3; (b) Central wire; (c) Wire 6.

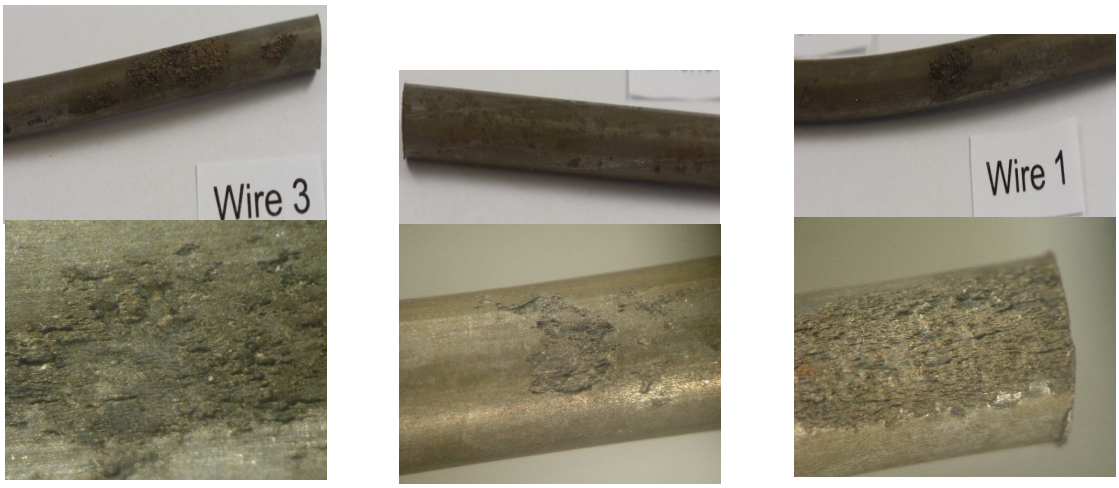


Figure 49. Wires from the strand coated with Civetea: (a) Wire 3; (b) Central wire; (c) Wire 1.

From Figure 38 to Figure 43, it can be observed that signs of corrosion were present even on some of the central wires after outdoor exposure (Trenton and Civetea); the corrosion extent was not as large as that observed on the outside wires. It is important to clarify that the pictures shown here were selected from the worst sites to show the most corroded parts. From the above observations, it can be deduced that corrosion initiated (via visual evidence) even on areas where there was a layer of the flexible filler. Sodium chloride or other types of particulates in the environment likely penetrated through the flexible filler to reach the metal surface and corrosion initiated in the steel wires. It is likely that moisture in the environment also played a role (due to high humidity, dew or rain). Appendix A shows additional detail.

3.3 Filler-coated Single-wire

3.3.1 Outdoor Exposure

Coated single-wire samples were exposed on May 5, 2016 (at the closed container and at the partially sheltered site on a shelf). Coated wires exposed at the partially sheltered site started to show rust on May 25, 2016 (Sanchem), (after 20 days). All other samples also started showing corrosion spots after longer exposure periods: Nontribos (43 days), Civetea (49 days), Trenton (114 days) and Sonneborn (126 days) (Figure 50 and Figure 51).

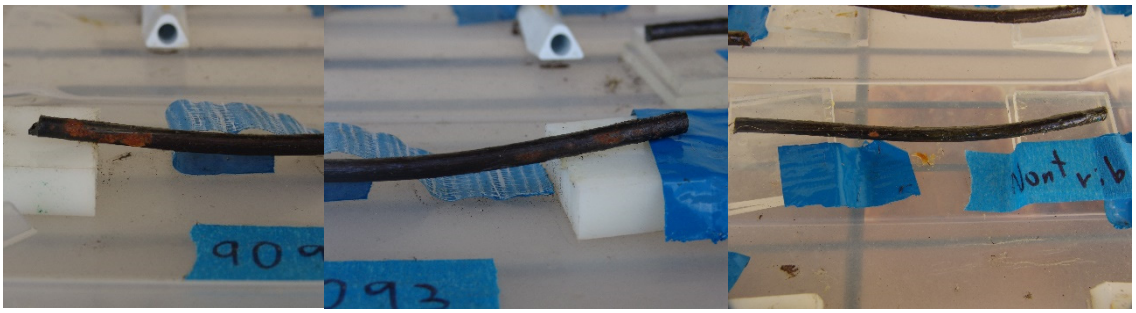


Figure 50. Wires coated and presence of rust. Left and Middle: Sanchem; Right: Nontribos, June 14, 2016.

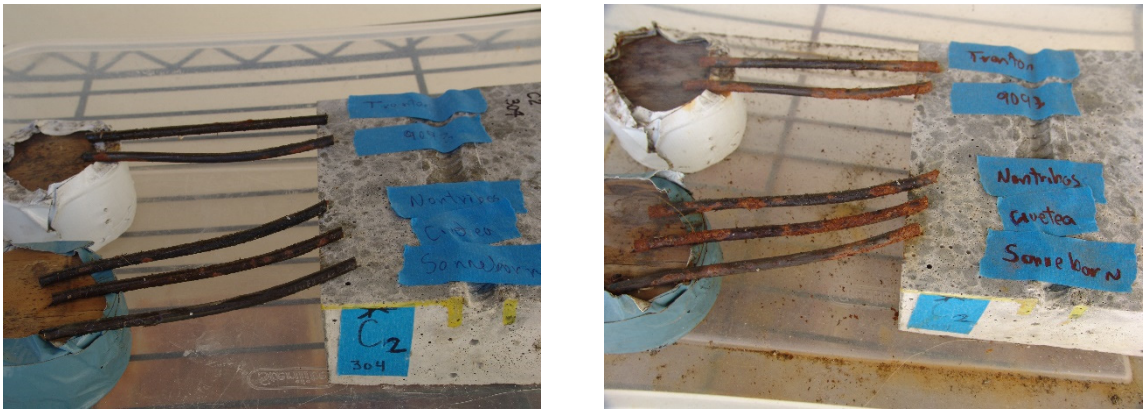


Figure 51. Wires coated with presence of rust. Left: July 28, 2016; Right: March 14, 2017.

By March 22, 2017 it was possible to see that rust spots were present in more than 50 % of the wire surface. Corrosion took place all around the wire. Exposure was terminated and the remaining filler was removed manually and with mineral spirits, followed by chemical cleaning. Finally, visual examination and then stereo microscope observations took place.

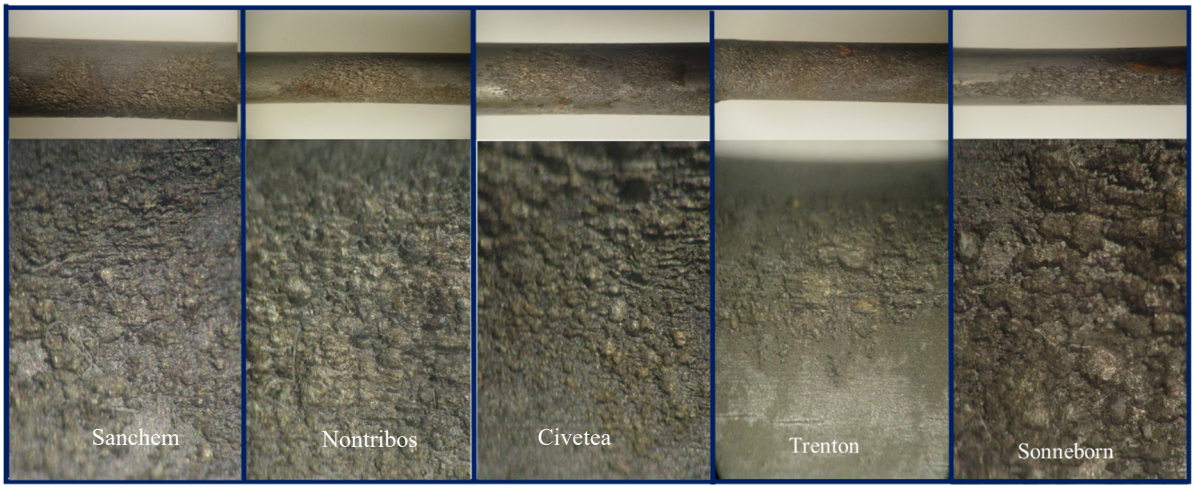


Figure 52. Microscopy of the samples outdoors at semi-sheltered location.

The stereo microscope pictures of the samples shown here focus on the sections that corroded the most. Figure 52 shows that general corrosion took place on all the specimens. The wire samples exposed outdoors in the closed container were cleaned with mineral spirits and an acid bath after 391 days of exposure and then were observed via stereo microscopy. The cleaned wires (Figure 53) did not present as much corrosion as the samples just described. These specimens exposed in the partially closed container presented rust on less than 8% of their total surface area. However, deep corrosion pits were not present(Figure 53).

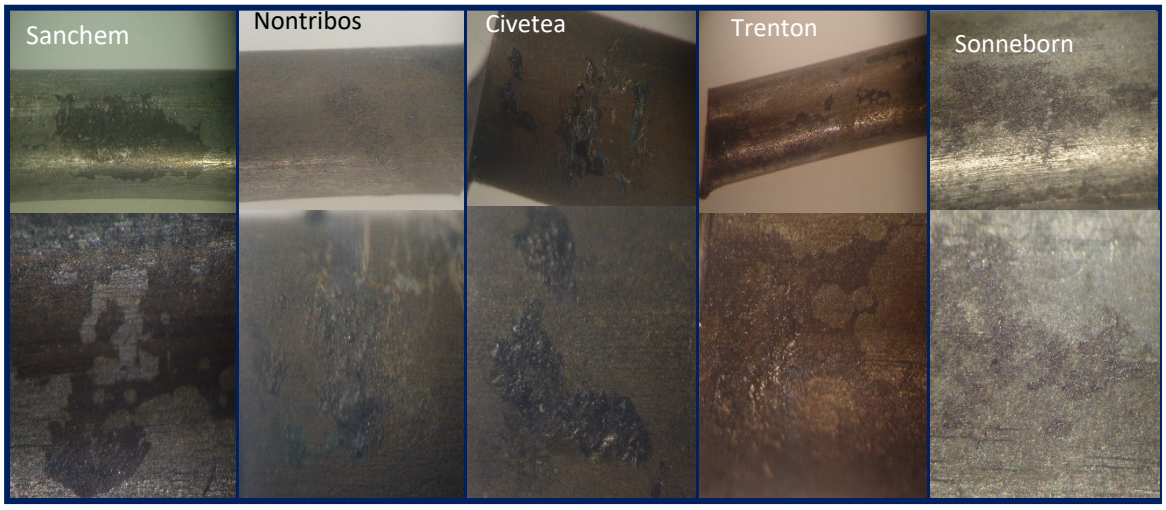


Figure 53. Microscopy of the samples partially closed container outdoors and high humidity.

3.3.2 Indoor Exposure

Laboratory samples exposed to high relative humidity and laboratory temperature (approx. 21°C) were exposed on May 25, 2016. Water was present below the wires at the mesh level. The non-coated wire presented rust 8 days after exposure. Coated wire samples started presenting rust after 19 days (Civetea). Before removing the samples, all wires showed corrosion at the supporting site of the wire. It is speculated that this area may have a high concentration of moisture (due to water wicking in to the support). These wire samples were cleaned after 371 days of exposure. Figure 54(b) shows the wires after cleaning and the corrosion extent in the coated wires exposed to high humidity in the lab at 21°C.

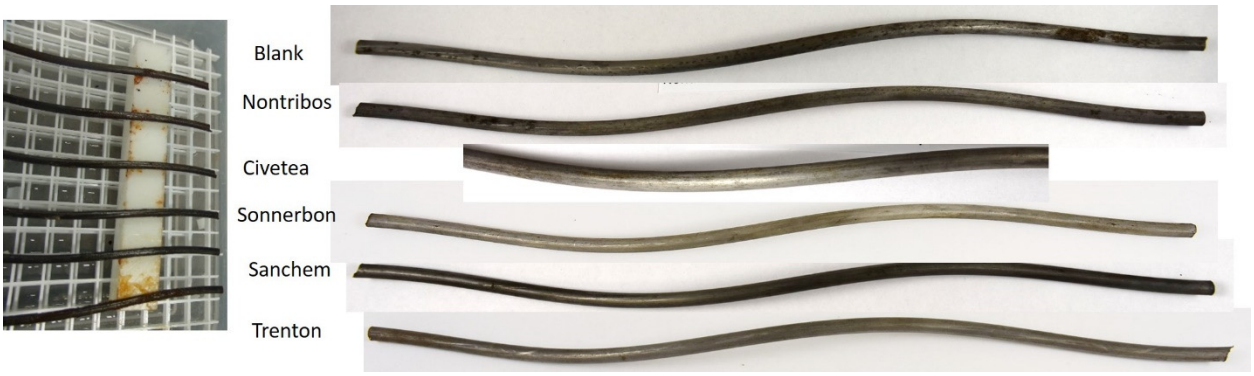


Figure 54. (a) Set up for the wires coated at lab; (b) Wires after cleaning.

After chemical cleaning, the samples were observed under the stereo microscope. From Figure 54(b), it is possible to observe that the non-coated wire is the one with the most corrosion, followed by Nontribos, Civetea, Sanchem, Trenton and Sonneborn. None of the wires exposed in this environmental condition presented rust on more than 30% of their surface area. Pictures of the samples shown in Figure 49 and Figure 50, were mainly focused on the sections with the most corrosion. Even though the coated wire specimen that presented a corrosion spot first is the wire coated with Civetea, it is not the coated wire with the most corrosion. It is possible that O_2 or H_2O

particles reach the wire coated with Civetea first. Another, possibility is that the filler layer was thinner. Corrosion did not propagate on the wire coated with Civetea as fast or as much as the corrosion observed on the wire coated with Nontribos or Sonneborn. Figure 55 and Figure 56 shows selected stereo microscopy views of these samples.

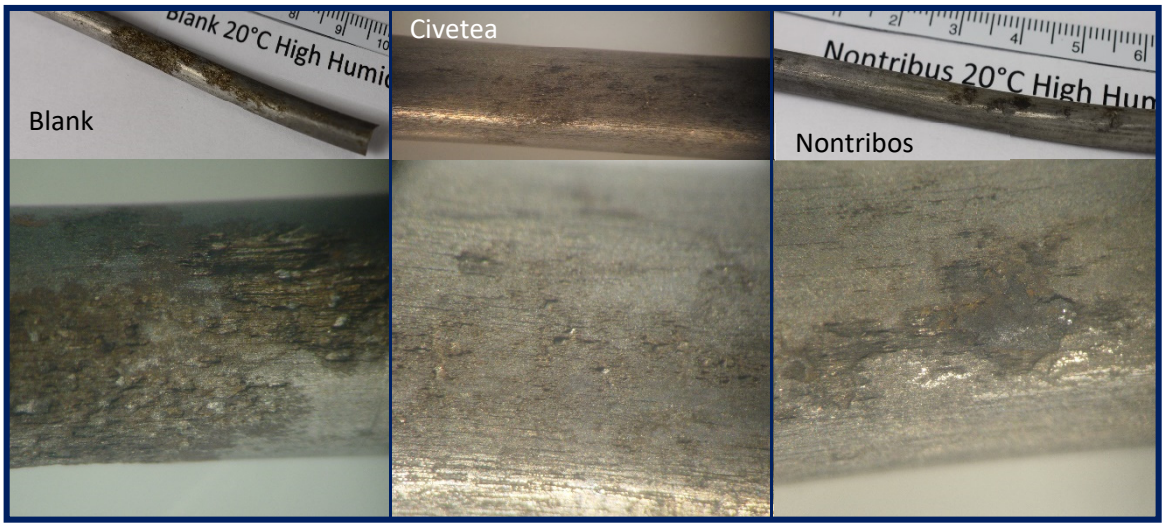


Figure 55. Microscopy of the samples in the lab at 21°C

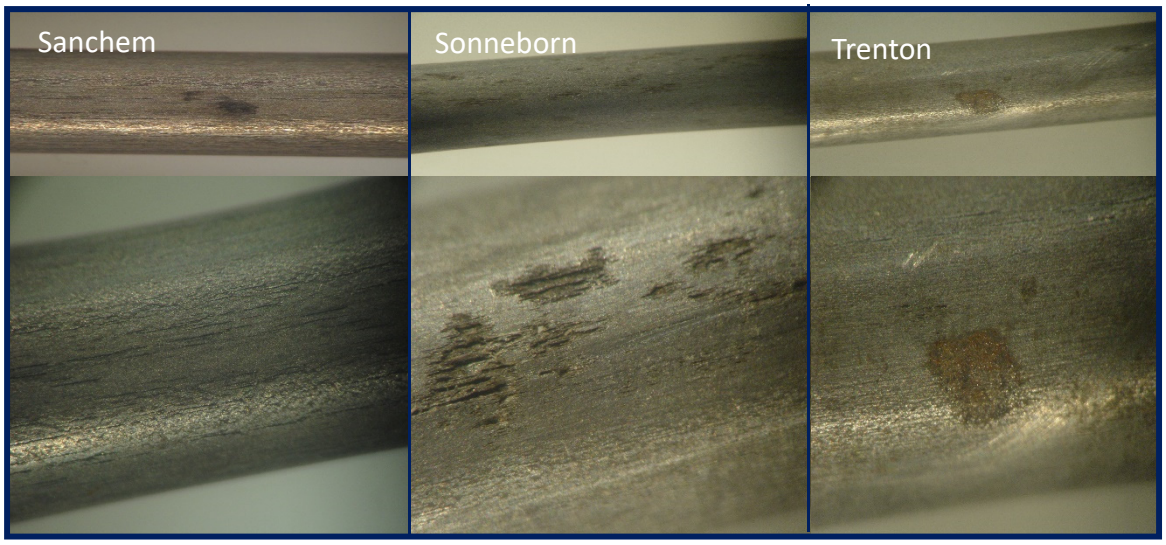


Figure 56. Microscopy of the samples in the lab at 21°C C

Samples exposed in high humidity at 32°C started exposure on November 16, 2016. The uncoated specimen presented rust after 16 days of exposure. Coated samples started presenting rust spots after 58 days (Civetea). By June 26, 2017, all the samples had corrosion spots visible at the site supporting the wire. Additional corrosion spots were observed in other areas only on the wires coated with Civetea and Sanchem. This set of wires were cleaned after 222 days of exposure. Figure 57 shows the wires before cleaning (left) and the blank wire, wire coated with Civetea, and wire coated with Nontribos after cleaning (right). Figure 58 and Figure 59 show close-ups after cleaning for each of the wires exposed in the elevated temperature condition.

Figure 57. Wires coated at elevated temperature room.

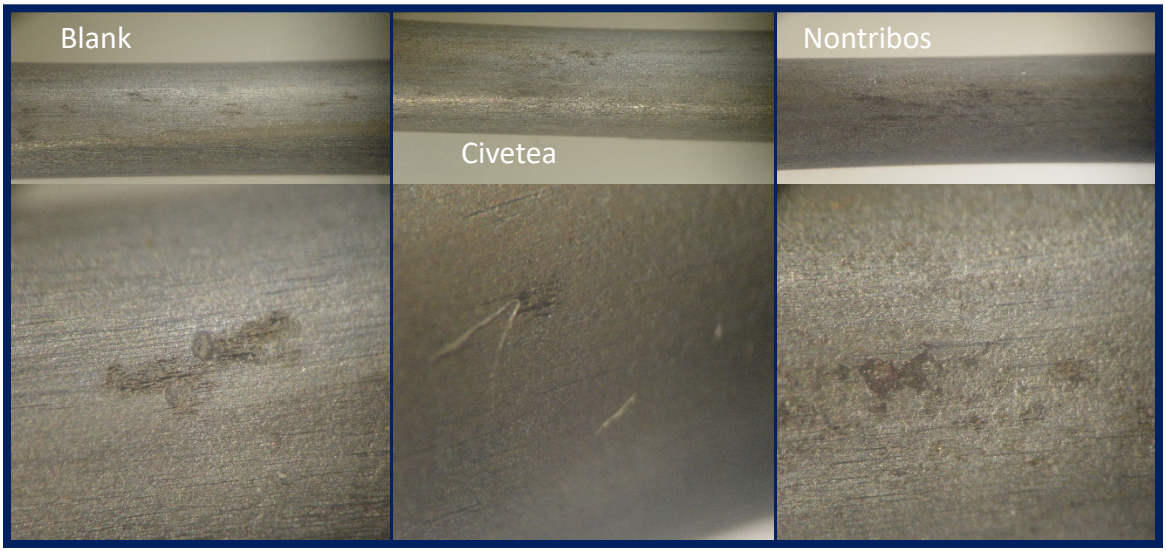


Figure 58. Microscopy of the samples in the elevated temperature room at 32°C.

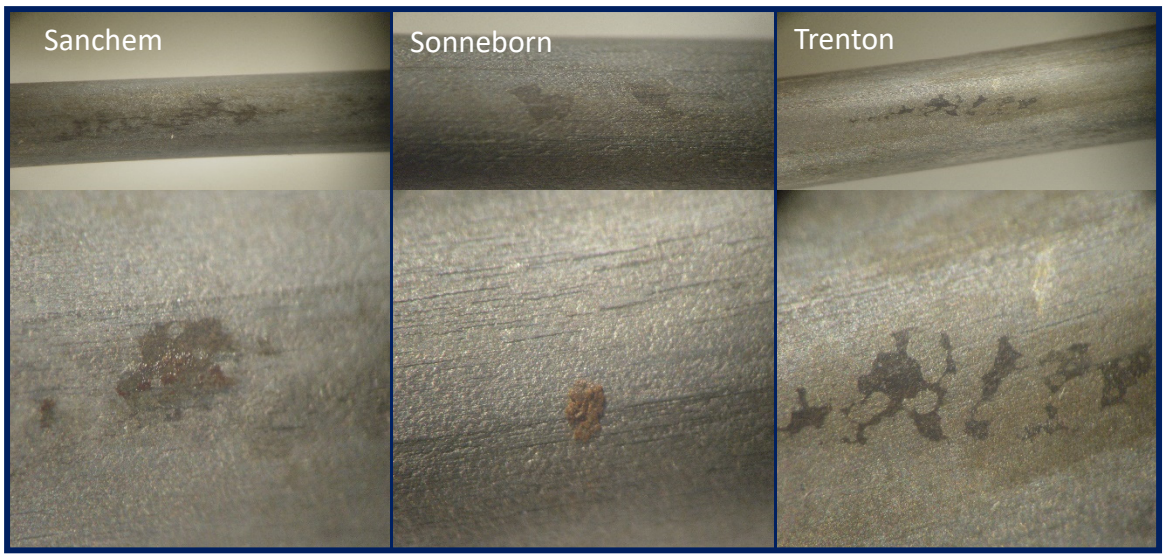


Figure 59. Microscopy of the samples in the elevated temperature room at 32°C.

In three out of four of the environmental conditions tested, the wire specimen that presented less corrosion was the wire coated with Sonneborn. With respect to the wire sample showing the most corrosion, (not including the blank wires), the wire coated with Nontribos showed the most corrosion. Table 17 summarizes the results after cleaning the wires exposed both indoors and outdoors.

Table 17. Corrosion Extent Single wire Filler-coated Summary.

Single wire coated				
Environmental condition	Time of exposure	% Exposure	Specimen most corrosion	Specimen with less corrosion
Partially closed container	321	82.1	Nontribos	Sonneborn
Outdoor shelve	391	100	Nontribos	Sonneborn
Lab @ 21°C High humidity	371	94.9	Nontribos	Trenton
ETR @ 32C High humidity	222	56.8	Nontribos	Sonneborn

It's important to note that the coated wires exposed to the different environmental conditions lasted different periods of time. Therefore comparing or ranking between exposure might not be possible. However, the results give an idea of how these environmental conditions induce corrosion on the coated wires. The samples exposed at the semi-sheltered location were exposed directly to chloride particles and other type of particulates present in the air as well as outdoor temperature and humidity. Appendix B shows additional forensic detail.

3.4 Filler-coated Single Wire Contaminated with Fungi

The exposure of inoculated wire samples sprayed with 1.5 milliliter started on December 10, 2016. Some wire specimens that were exposed in the lab at 21°C presented rust on January 13, 2017 (34

days). This was observed via visual inspection due to the change in color. All samples (hoses) appeared to have some solution. The excess solution was removed from all samples and stored in small vials shortly after day 34.

After visual inspection, all Nontribos coated samples and all specimens sprayed with *Fusarium oxysporum* were selected to be opened. This took place on January 25, 2017 (44 days after exposure started). The rest of the solution inside the transparent tubing was stored in small containers. The remaining layer of filler was removed and stored for future analysis. The coated wire specimens were then cleaned. The wire samples were observed using a stereo-microscope (Figure 60).

For wires coated with Nontribos, the wire samples sprayed with the fungi mix displayed the most corrosion, followed by the specimen sprayed with *Fusarium oxysporum* (Figure 60),. The sample sprayed with water showed corrosion but to a lesser extent. From the samples coated with the various fillers and inoculated with *Fusarium oxysporum* (Figure 61), the wire that presented more corrosion was the wire coated with Nontribos, followed by the wire coated with Sanchem .The sample with the least corrosion was the wire coated with Sonneborn.

For the rest of the specimens the remaining solution was removed the day that the wire samples coated with Nontribos and the wires sprayed with *Fusarium oxysporum* were opened. The samples continued exposure in the laboratory environment. The rest of the wire samples were cleaned on May 31, 2017 (170 days of exposure).

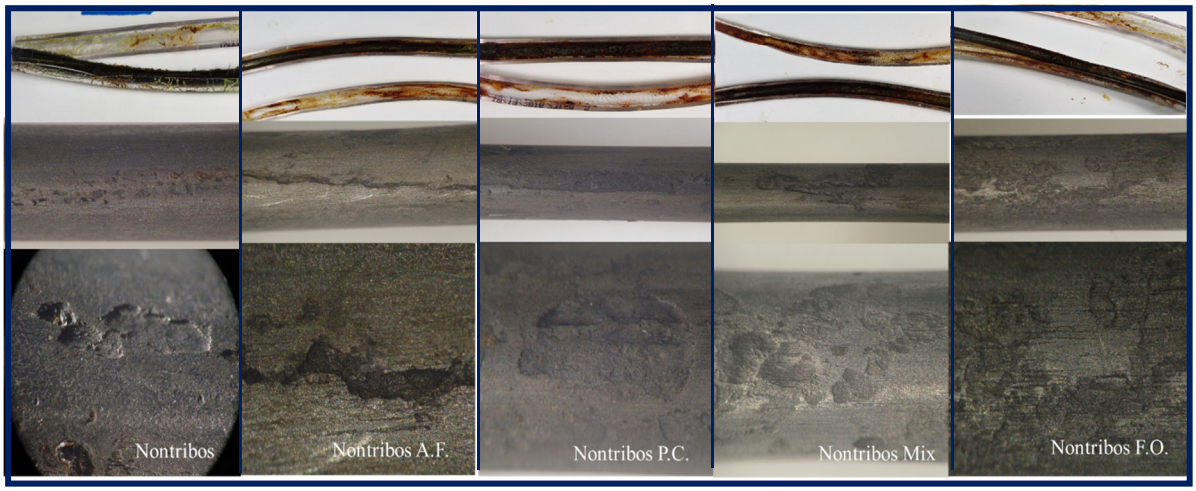


Figure 60. Microscopy of wires coated with Nontribos and inoculated with fungi

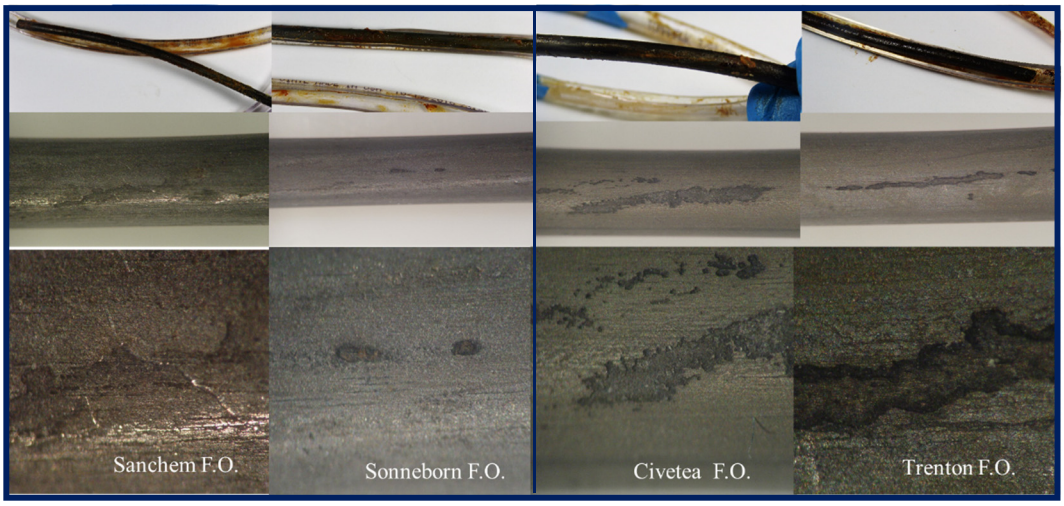


Figure 61. Wires coated as indicated and inoculated with *Fusarium oxysporum*.

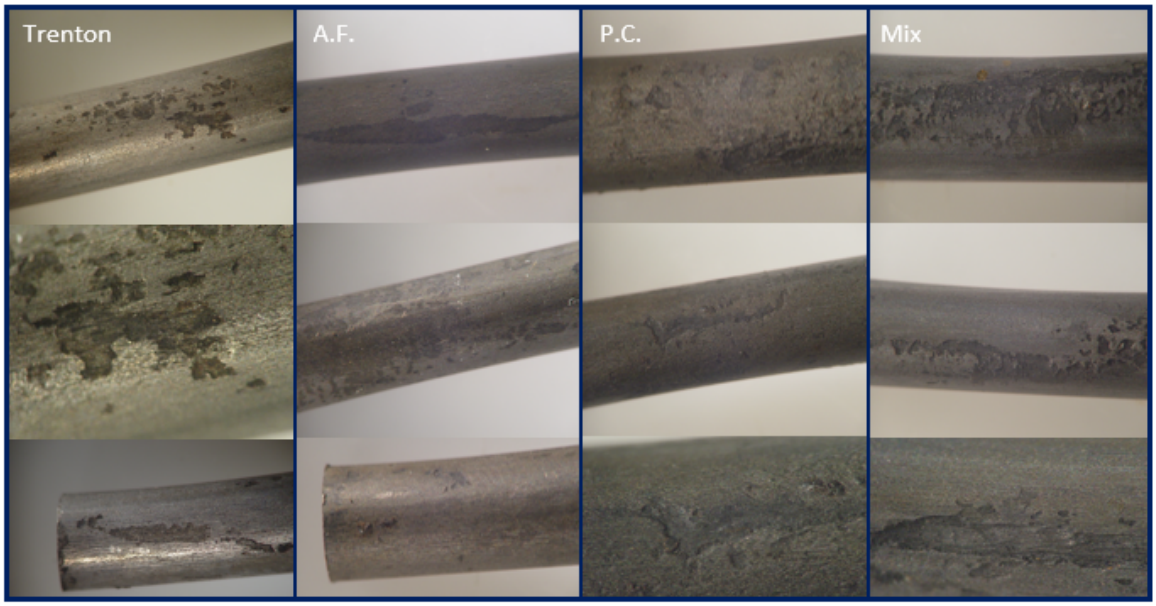


Figure 62. Wires coated with Trenton.

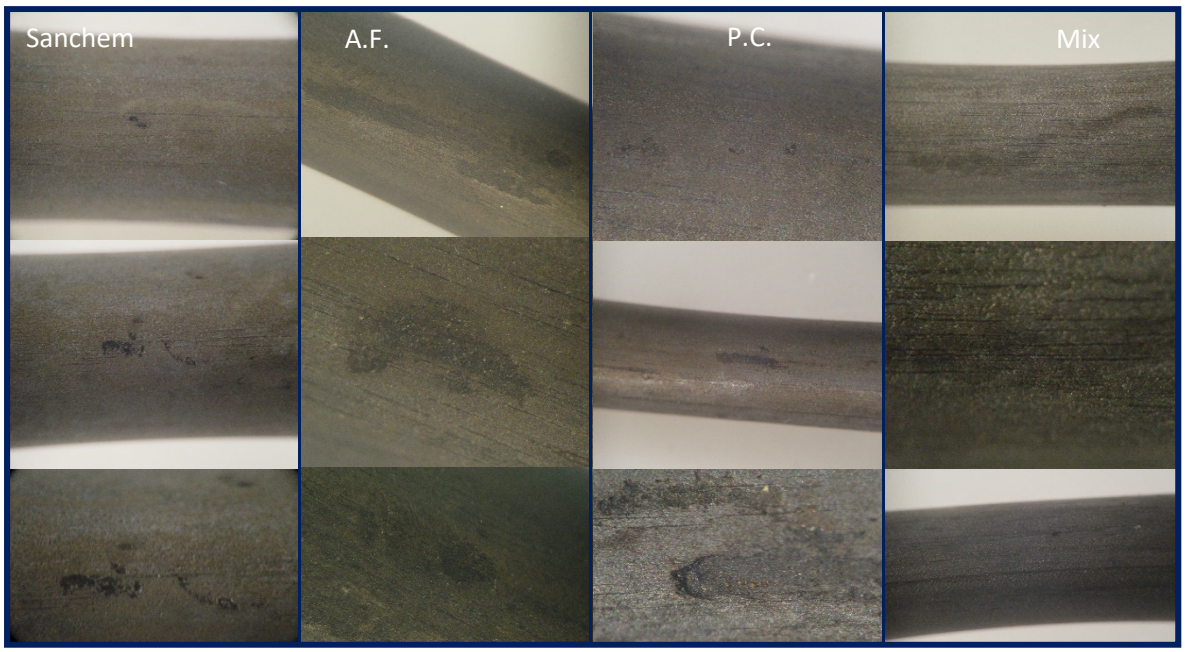


Figure 63. Wires coated with Sanchem.

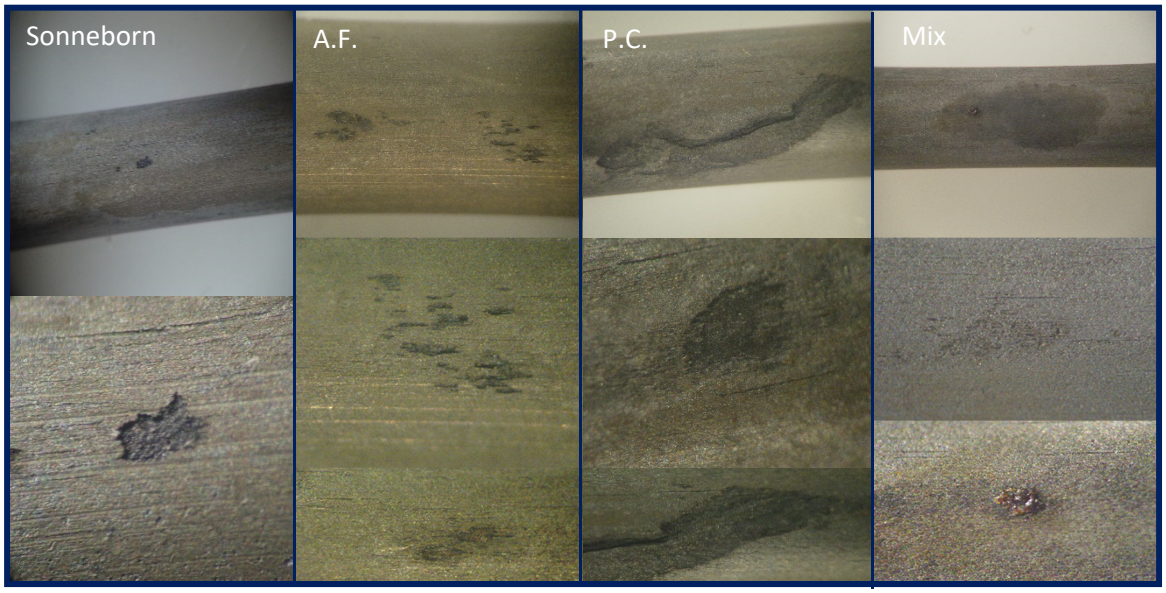


Figure 64. Wires coated with Sonneborn

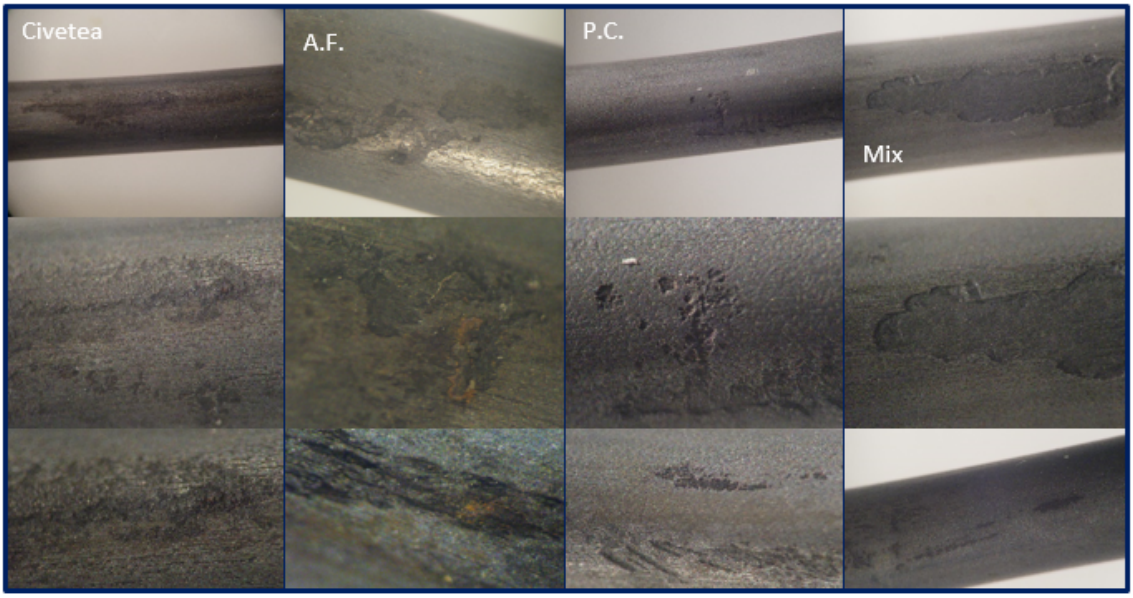


Figure 65. Wires coated with Civetea.

For the remaining wires coated with flexible fillers (Trenton, Sanchem, Sonneborn and Civetea) Figure 62 (Trenton), Figure 63 (Sanchem), Figure 64 (Sonneborn), and Figure 65 (Civetea) show in three sets that the coated wire specimens sprayed with the fungi mix presented the most corrosion. This trend was similar to the case of the wire samples coated with Nontribos. The wires coated with Sonneborn presented the most corrosion on the wire sprayed with the *Penicillium chrysogenum* suspension. It is possible that some of the solution stayed in the hose for this wire, increasing the moisture which then led to additional corrosion. It could also be possible that the layer of the flexible filler was thinner in some parts of the wire or even removed during the process of introducing the wire into the transparent hose.

From Figure 60, Figure 62, Figure 63, Figure 64, and Figure 65, it can be observed that the samples sprayed with water show less corrosion than the samples sprayed with fungi (wire samples sprayed with water are those on the left in these figures). With respect to the samples sprayed with one type of fungi, it is possible to see that the specimens sprayed with *Fusarium oxysporum* presented the most corrosion, followed by the samples sprayed with *Aspergillus flavus* and finally the wires sprayed with *Penicillium chrysogenum*. These results are consistent with the results obtained in the first section (inoculated fillers), where *Fusarium oxysporum* and *Aspergillus flavus* were able to use the flexible fillers as a carbon source and developed on them. Appendix C shows additional forensic detail.

3.4.1 Samples Sprayed with Fungi (Smaller Volume Set 2)

The second batch of wires that were coated by dipping in the melted filler, placed in a transparent hose and then sprayed with 0.6 milliliter of suspension (similar than described in the above section) were terminated during July 2017. Appendix D shows additional detail than what is presented below. The exposure for these samples started on February 15, 2017 and were cleaned on July 18, 2017, thus the exposure lasted for 153 days. Significantly less corrosion extent was observed on

the wires sprayed with 0.6 milliliter of suspension with fungi upon cleaning compared to the wires sprayed with 1.5 milliliter of suspension with fungi. The better performance could, in part be explained by the method used to apply the filler onto the wire.

Figure 66 shows the wire coated with Trenton and sprayed with fungi mix. The pictures show the wire after cleaning. There were a number of corrosion spots on this wire. Figure 67 shows sections of the wires coated with the other fillers and sprayed with fungi mix. Less corrosion was observed on these wires compared to the wires shown in Figure 60.

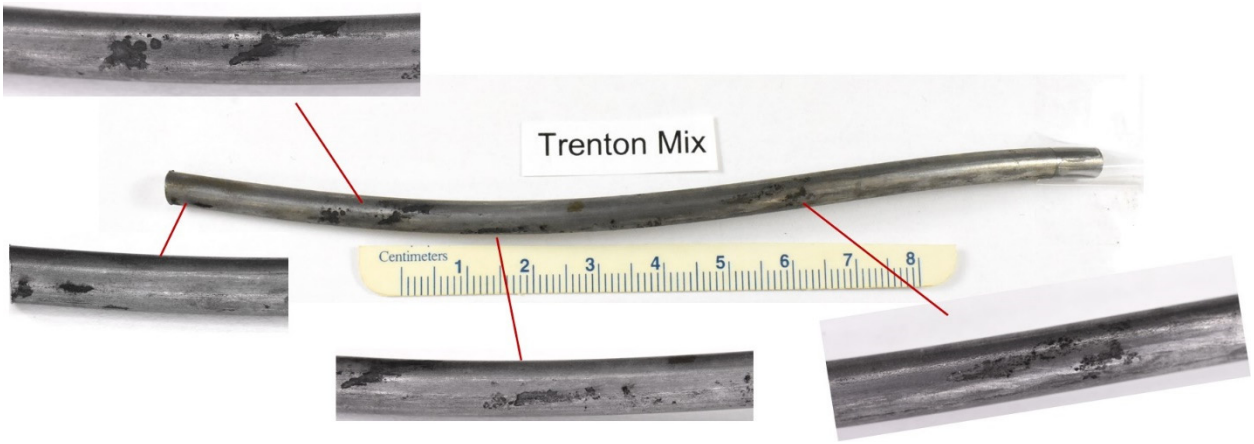


Figure 66. Wires coated with Trenton and sprayed with fungi mix after cleaning.

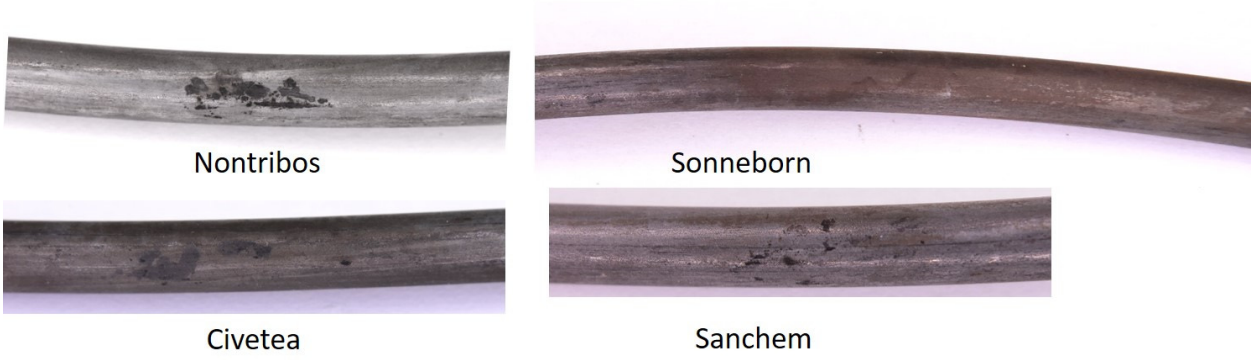


Figure 67. Wires coated with the other fillers and sprayed with fungi mix after cleaning

.

Figure 68 shows the wire coated with Trenton and sprayed with fungi *Penicillium chrysogenum*. The pictures show the wire after cleaning. Upon rotating the wire, there were other corrosion spots observed. Figure 69 shows two close-ups and two pictures taken with the stereo microscope from two sites on the same wire shown in Figure 68. A modest amount of cross-section loss is observed.

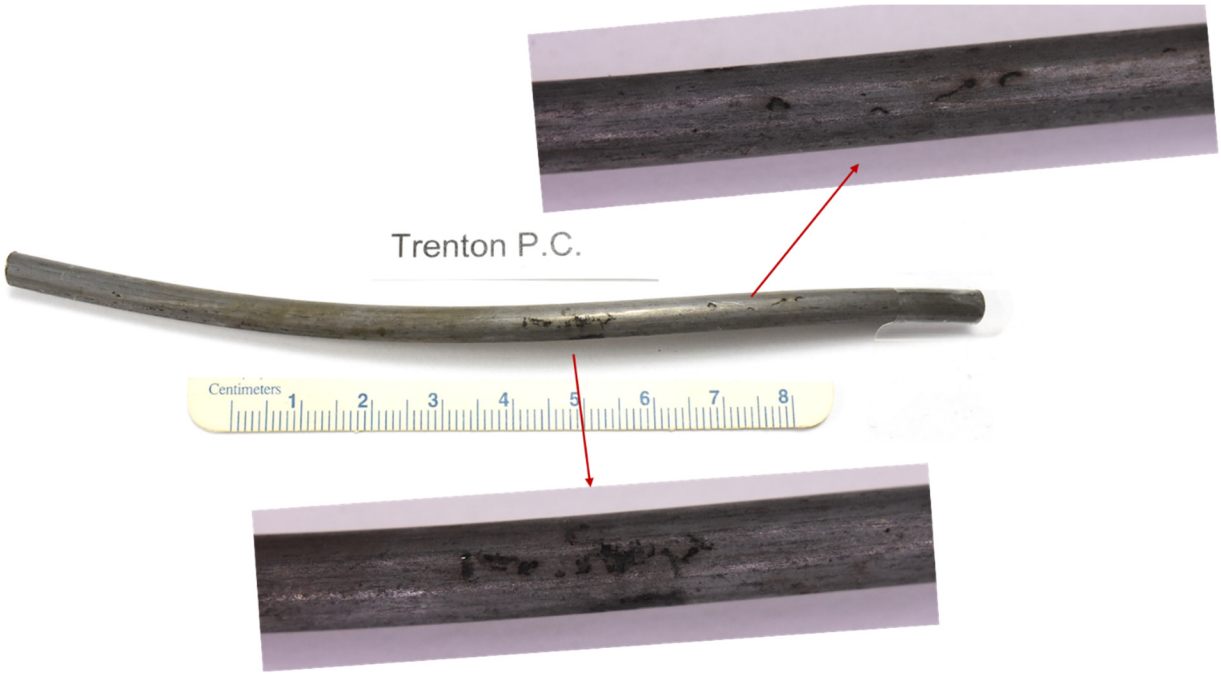


Figure 68. Wires coated with Trenton and sprayed with *Penicillium chrysogenum* after cleaning.

Figure 69. Close-ups – wire coated with Trenton and sprayed with *Penicillium chrysogenum*.

3.5 Electrochemical Cell

3.5.1 Strand Coated with Civetea

Figure 70 shows the evolution of impedance magnitude and galvanic current of the steel strand coated with Civetea filler in a simulated soil solution with different pH values as a function of exposure time. The impedance magnitude is obtained at a frequency of 1 Hz, and the galvanic current is the current between the steel strand electrode and the magnesium and copper electrode which were obtained at a time of 100 seconds of being coupled. It can be observed that at around 16 days, there is an increase in the galvanic current and a decrease in the impedance magnitude with respect to the magnesium electrode. The sudden change in both the impedance magnitude and the galvanic current is attributed to the corrosion initiation at the strand steel and/or at the magnesium, which was caused by penetration of solution through the filler layer. This was further confirmed by the visual observation (Figure 65). However, there is no change with respect to the copper electrode. This is probably because the filler layer on the copper electrode was still intact, while the filler layer on the steel and magnesium electrode was not.

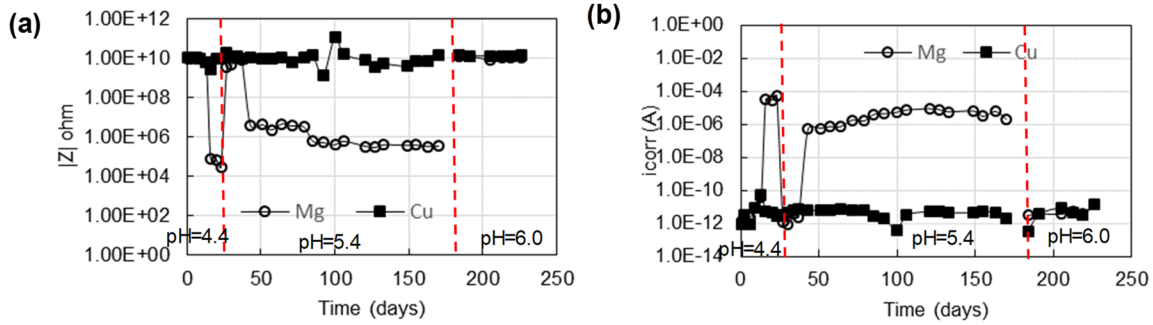


Figure 70: Evolution of impedance magnitude and galvanic current as a function of immersion time in solution with different pH values for steel strand coated with type 3 filler (Civetea).

After approximately 21 days of testing, the steel strand was taken out of the solution and visual examination on the surface was performed. Then the steel strand sanded and was placed back and the solution was replaced with a pH of 5.4. After approximately 16 days of immersion in the solution with a pH of 5.4 (at 37 days), a decrease in the impedance magnitude and an increase in the galvanic current were observed. This observation is related to another corrosion initiation event. The test continued and the cell was open by day 175 for visual inspection. After visual observation, the steel strand and the other electrodes were sanded coated with the filler and then placed back on the cell and the solution was replaced with a pH of 6.0.

Figure 71 shows the surface conditions of all the electrodes after 21 days of immersion testing in soil solution with a pH=4.4. The steel strand electrode presented wires with corrosion (there are several wires with pits). The magnesium electrode presented a relatively large amount of corrosion to the left of the electrode, while the copper electrode did not present corrosion.

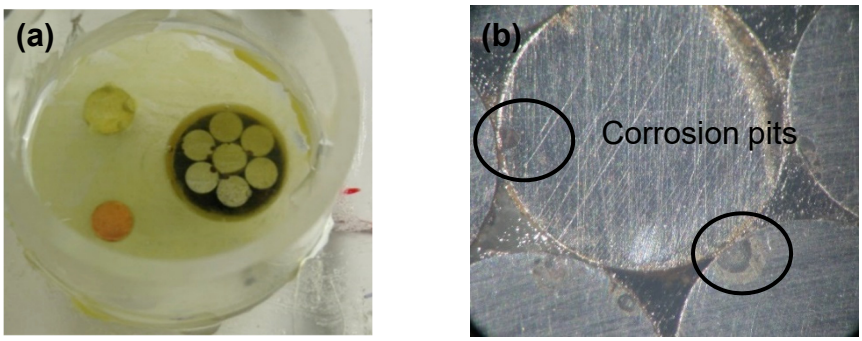


Figure 71: Visual observation of electrodes coated with type 3 filler after 21 days of immersion in a pH of 4.4 solution.

Figure 72 shows the surface conditions of all three electrodes after immersion testing in pH 5.4. No significant corrosion was observed on the steel strand, while the magnesium showed signs of corrosion. As for the copper electrode, no corrosion was present.

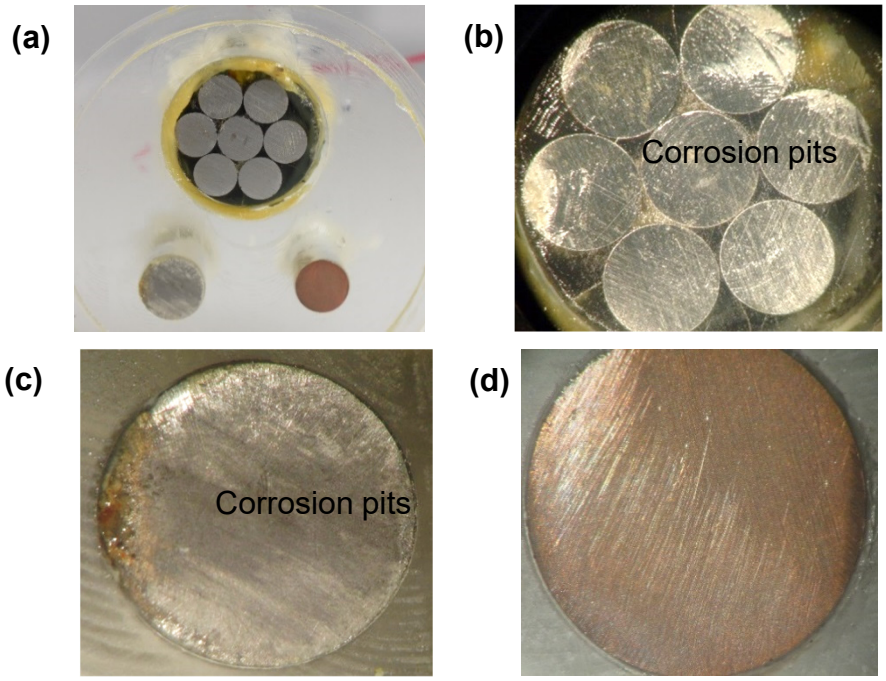


Figure 72: Surface conditions of the electrodes coated with type 3 filler (Civetea) in a pH of 5.4 solution (at day 175).

3.5.2 Filler Type 4: Trenton

Figure 73 shows the evolution of impedance magnitude and galvanic corrosion current for steel strands coated with Trenton filler and immersed in soil solution with different pH values. At the time of 175 days, a sudden drop in the impedance magnitude and a sudden increase in the galvanic corrosion current were observed, due to the penetration of test solution through the filler layer to the substrate steel strand, magnesium, and copper electrodes. This resulted in the initiation of corrosion pits. This was confirmed by visual observation after opening the specimens.

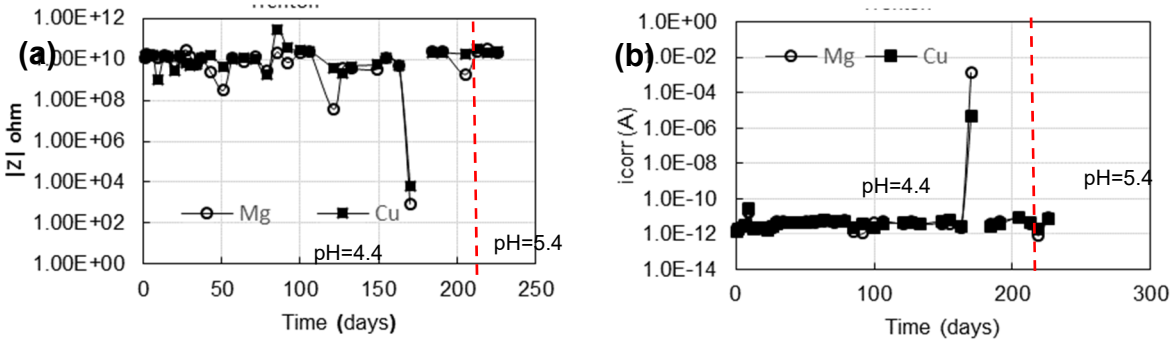


Figure 73: Evolution of impedance magnitude and galvanic current as a function of immersion time in solution with different pH values for steel strand coated with type 4 filler (Trenton).

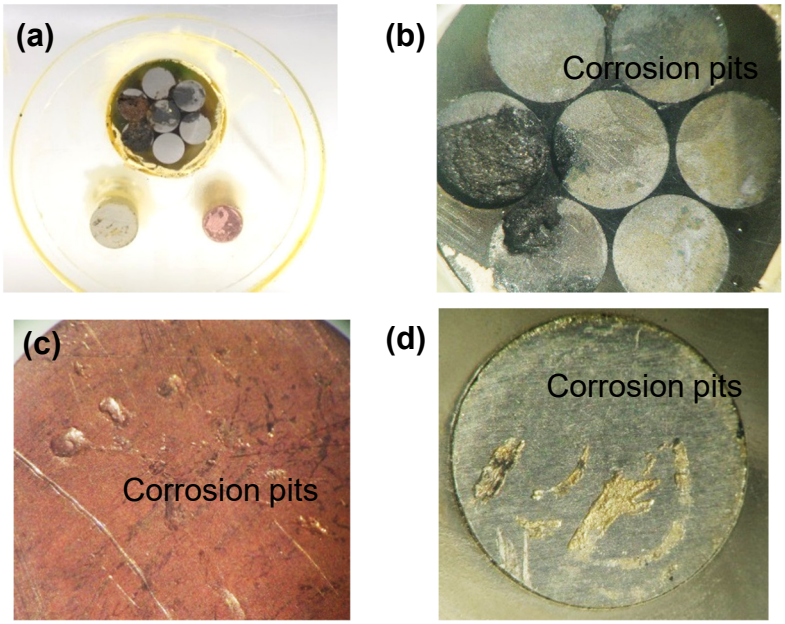


Figure 74: Visual observation of electrodes coated with type 4 filler (Trenton) after 175 days with a 4.4 pH solution (a) Electrodes, (b) Steel strand, (c) Copper electrode, and (d) Magnesium electrode.

Figure 74 shows the surface condition of electrodes coated with type 4 filler after immersion in a 4.4 pH solution for 175 days. It can be seen that four out of the seven-wires in the steel strand displayed corrosion. The corrosion pits were deep (around 4.0 millimeters). For the copper and magnesium electrodes, there were pitting holes on the surface. For the copper electrode, the depressions were more localized, while for the magnesium electrode three large deep corroding sites and a large number of small pits were present.

3.5.3 Filler Type 5: Sonneborn

Figure 75 shows the evolution of impedance magnitude and galvanic corrosion current for steel strands coated with type 5 filler and immersed in soil solution with different pH values. After approximately 140 days of immersion testing, a drop in the impedance magnitude and an increase in the galvanic corrosion current are observed with respect to both copper and magnesium

electrodes. This is attributed to corrosion initiation after penetration of solution through the filler layer.

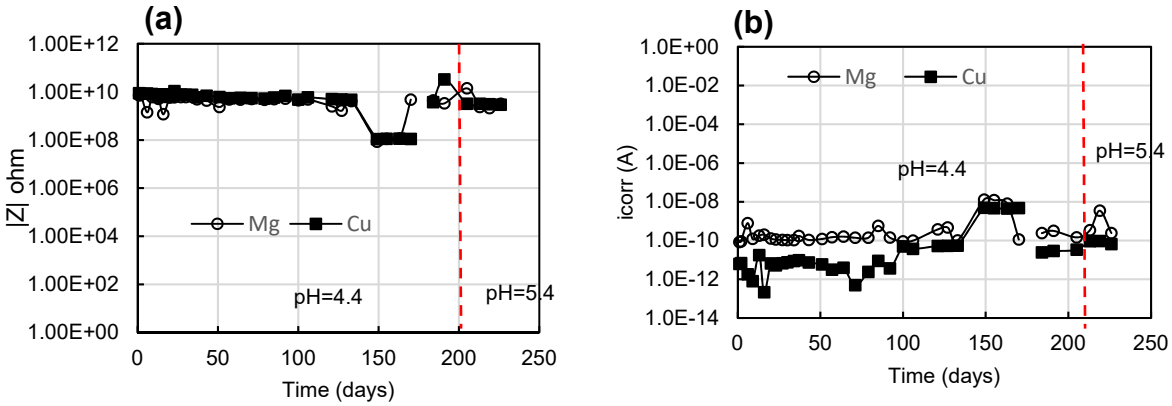


Figure 75: Evolution of impedance magnitude and galvanic current as a function of immersion time in solution with different pH values for steel strand coated with type 5 filler (Sonneborn) .

Figure 76 shows the surface conditions of the electrodes after immersion in 4.4 pH solution for 175 days. Obvious pitting corrosion is observed on all three electrodes. For the steel strand, only one wire showed corrosion, while the rest of the wires did not display any corrosion. A deep corrosion pit is observed on the magnesium electrode and several corrosion marks were observed on the copper surface.

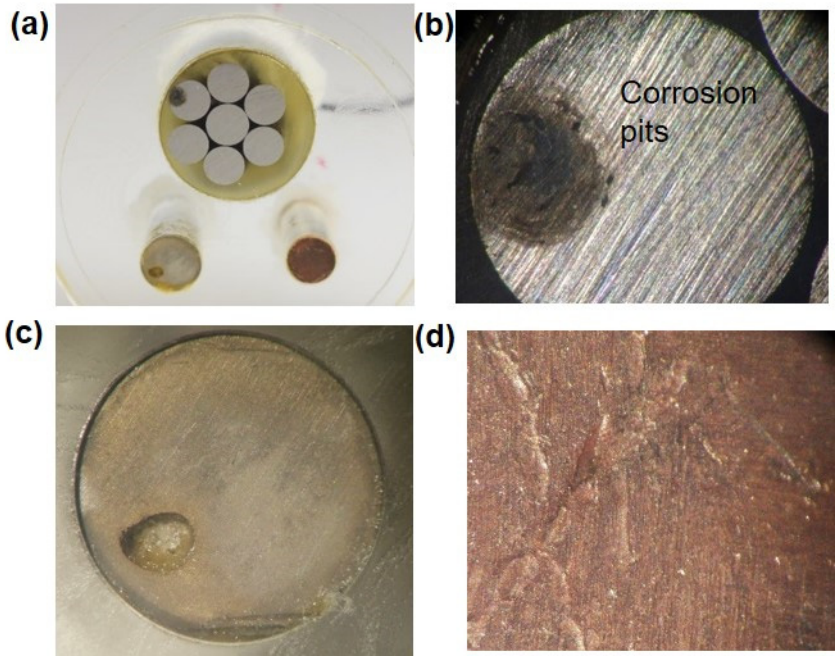


Figure 76: Visual observation of electrodes coated with type 5 filler (Sonneborn) after 175 days with a 4.4 pH solution (a) Electrodes, (b) Steel strand, (c) Magnesium, and (d) Copper electrode.

3.5.4 Filler Type 1:Sanchem; and Filler Type 2 :Nontribos

Figure 77 shows the change of impedance magnitude and galvanic corrosion current for steel strand electrodes coated with Sanchem (type 1) and Nontribos (type 2) fillers as a function of time in a 4.4 pH solution. These were different from the other three fillers. There was no change in both the impedance magnitude and galvanic current over time for the electrochemical cells with electrodes coated with Type 1 and Type 2 fillers. The impedance magnitude remained around 10 GΩ and the galvanic corrosion current was around 1.0 pA. The high impedance magnitude and the low galvanic corrosion current indicate that both type 1 and type 2 fillers have better corrosion protection compared with the other three fillers.

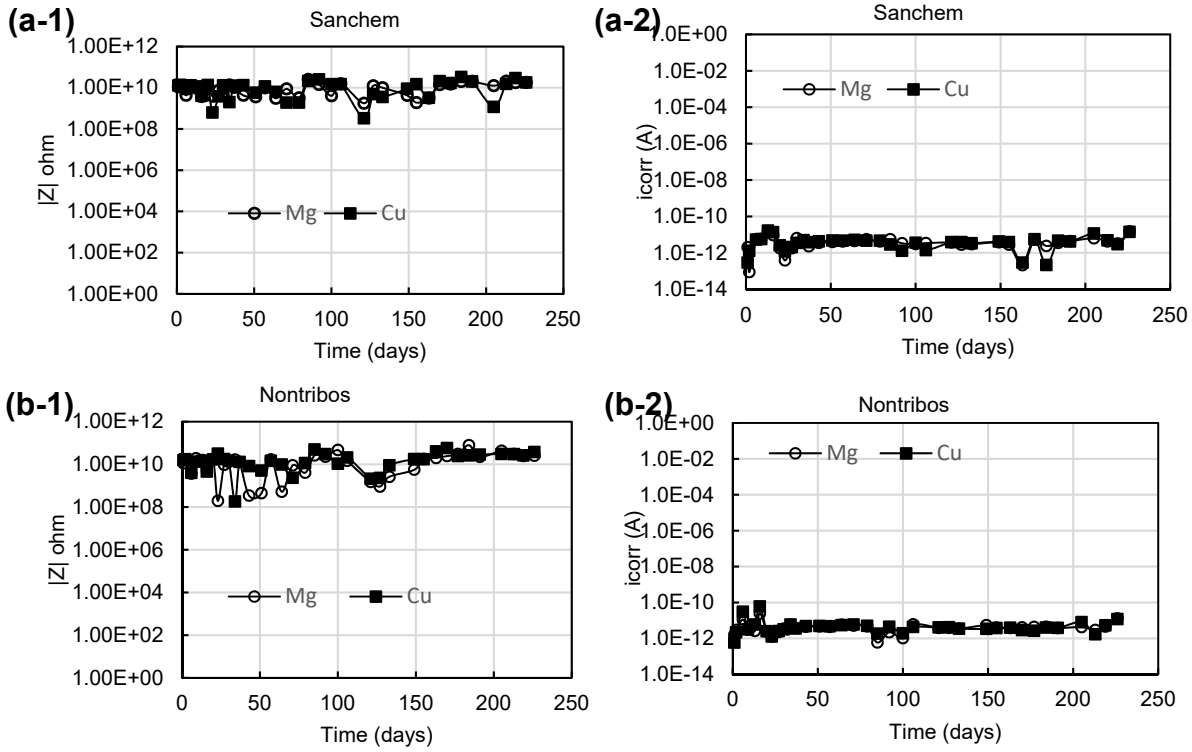


Figure 77: Evolution of (1) impedance magnitude and (2) galvanic current for steel strand electrodes coated with (a) type 1 (Sanchem) and (b) type 2 (Nontribos) in pH=4.4 solution over time.

Chapter 4 - Larger Samples

4.1 Visual Inspection of Larger Specimens

As indicated in the experimental section, visual inspections were performed periodically on the larger samples. Additionally, pictures were taken after removing the caps (in some cases pictures were taken from both ends, but not always). The following paragraphs will describe the surface condition of the samples upon removing the caps during the inspection carried out during December 2017 and a more recent inspection that took place during May or June 2018.

4.1.1 Four-inch Diameter Samples Injected with Trenton.

No changes based in sample appearance were identified on the samples that were not sprayed. Figure 78 shows pictures of both ends for a sample sprayed with water (the picture on the right shows the end sprayed with water) for the inspection that took place during December 2017. Figure 79 shows a picture of the side sprayed with water during the inspection that took place during June 2018. No significant changes were observed when comparing the pictures from December 2017 and May/June 2018. It appears that some corrosion (as suggested by rust spots) has taken place on a few wires on the side sprayed with water. These wires might have had a thin layer of the filler, whereas a number of strands were protected because they were covered with a thicker layer of the Trenton filler. Figure 80 shows the surface condition for the Sample Sprayed with fungi mix during the visual inspection that took place on December 2017. Figure 81 shows the surface sprayed with fungi mix during the inspection taken May 2018. Droplets of solution are visible in the picture on the right side of Figure 80, but not in Figure 81. The difference could be the time of the inspection with respect to the time the sample was sprayed and/or also that the temperature during May/June are significantly higher and it is likely the solution evaporated faster. It is not clear if the dark green surface is due to the cut, the fungi reacting with the filler or if it is a combination of both the cut and the fungi.

Not Sprayed – Bottom Side

Sprayed with Water

Figure 78. Four-inch diameter sample sprayed with water (December 2017).

Figure 79. Four-inch diameter sample, side sprayed with Water (June 2018).

Not Sprayed – Bottom Side

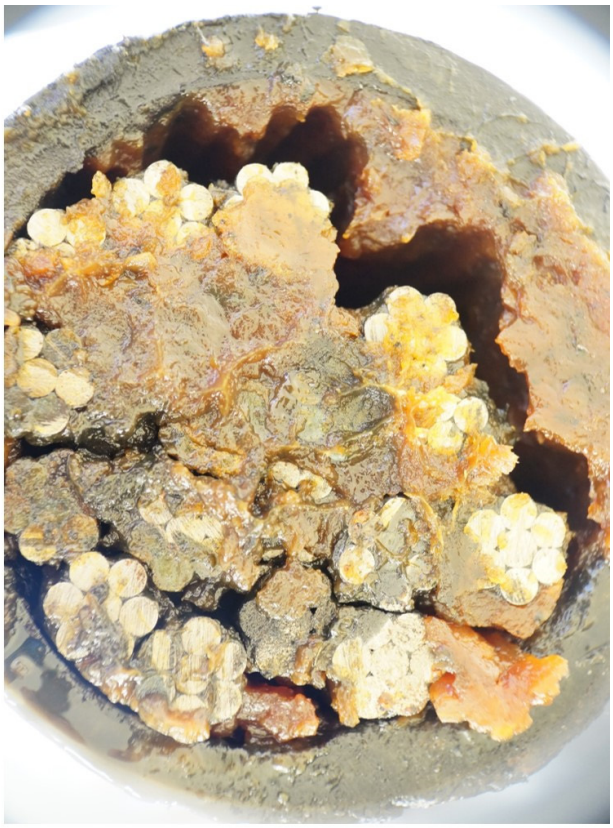
Sprayed with Fungi Mix

Figure 80. Four-inch diameter sample sprayed with fungi mix (December 2017).

Figure 81. Four-inch diameter sample, side sprayed with fungi mix. June 2018.

Figure 82 to Figure 87 show the surface condition for the samples initially sprayed with FO, PC and AF, respectively. The spraying that took place at a later time was done with water. Figure 82, 84, and 86 show a picture of the surface on the right that was initially sprayed with the fungi solution. Figure 82, Figure 84 and Figure 86 show pictures taken during December 2017, and Figure 83, Figure 85 and Figure 87 show pictures taken during May/June 2018. Based on what the pictures showed. It is not clear if the fungi has spread or if corrosion has initiated. There are a few strands that have one or two wires that appear to be corroding, as indicated by rust stains. In most of the pictures showing the sprayed sides; there are gaps/separation between the filler and the strands. It is possible that some of the filler leaked-out during the cuts done to obtain shorter segments. It is also possible that during exposure some of the filler flowed downwards. The sample that appears to have the most corrosion is the specimen initially sprayed with AF (Figure 87).

Not Sprayed – Bottom Side



Sprayed with F.O.

Figure 82. Four-inch diameter sample sprayed with FO (December 2017).

Figure 83. Four-inch diameter sample initially sprayed with FO, side sprayed with water. (May 2018).

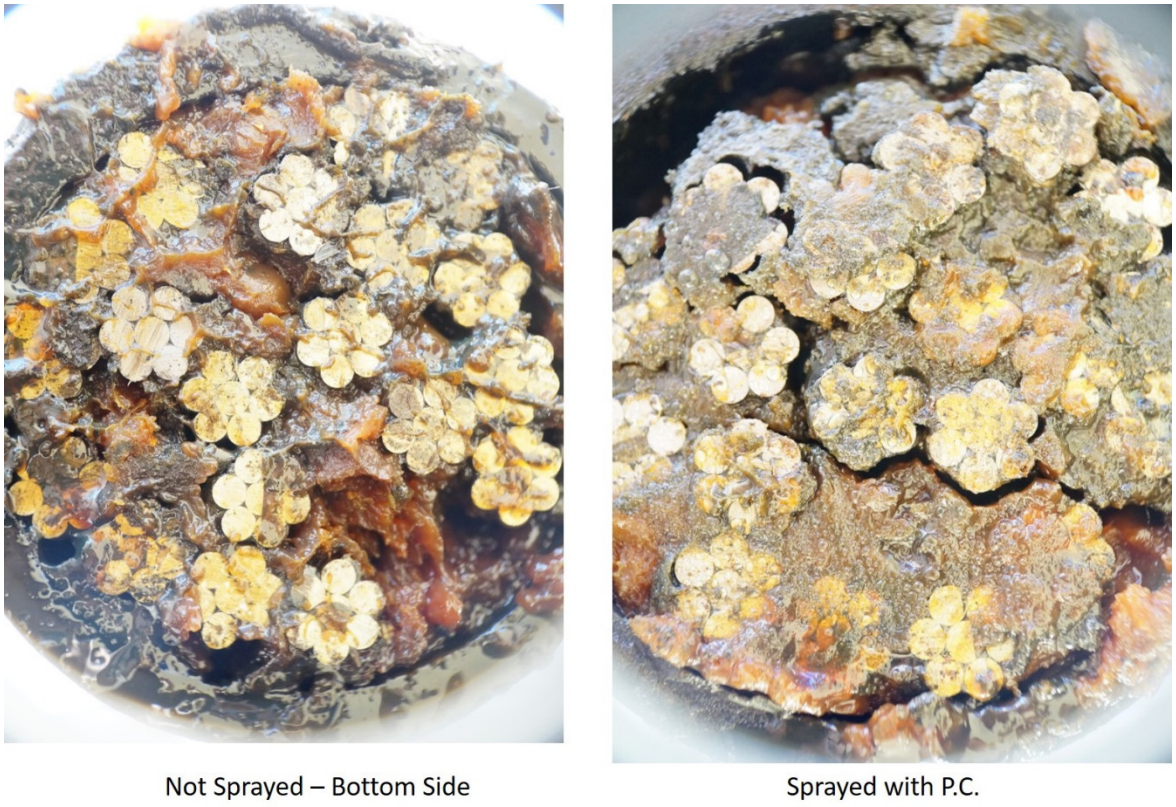


Figure 84. Four-inch diameter sample injected with Trenton and initially sprayed with PC. (December 2017)

Figure 85. Four-inch diameter sample Trenton and initially sprayed with PC, side sprayed with water. (June 2018).

Figure 86. Four-inch diameter sample injected with Trenton and sprayed with AF (December 2017).

Figure 87. Four-inch diameter sample injected with Trenton and initially sprayed with AF, side sprayed with water. (May 2018).

4.1.2 Two-inch HDPE Samples Injected with Various Fillers.

In this section only the samples sprayed with fungi will be described, starting with NC samples (no conditioning on the strands). Pictures of the samples sprayed with water are included in an appendix.

Figure 88 shows the surface condition after removing the caps on both ends of the NC samples injected with Civetea and sprayed with fungi mix (picture on the right) during December 2017. A slight change in color appears to have taken place, but it might be due to a camera and/or light effect. The strand that is visible does not appear to be corroding. Figure 89 shows similar pictures for the sample injected with Civetea and sprayed with fungi mix during an inspection that took place during June 2018. Two strands are visible in the picture on the left and one strand is visible in the picture on the right. No corrosion is visible in the pictures shown in Figure 89.

Figure 90 shows pictures of the Trenton injected NC sample and sprayed with fungi mix, for the inspection that took place December 2017. For this sample two strands are visible in the left picture with partial coverage of the filler. The picture on the right (side sprayed) only shows Trenton filler and no strand is visible. No color change is apparent. Figure 91 shows similar picture for the inspection performed during June 2018. The picture on the left shows two strands with good filler coverage and there is a thicker layer.

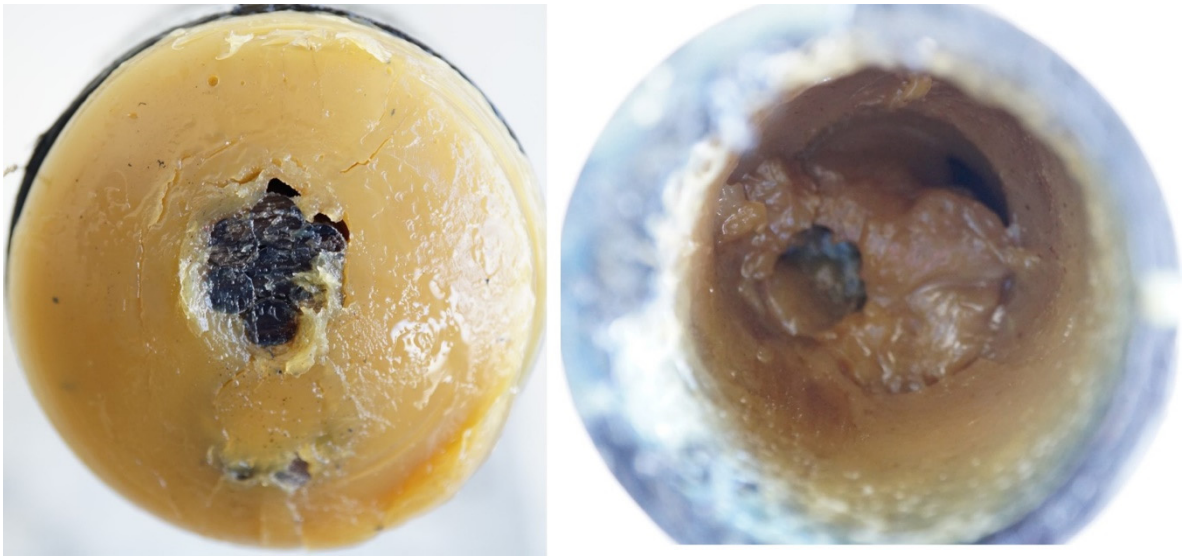


Figure 88. Civetea NC sample sprayed with fungi mix (one side) (December 2017).

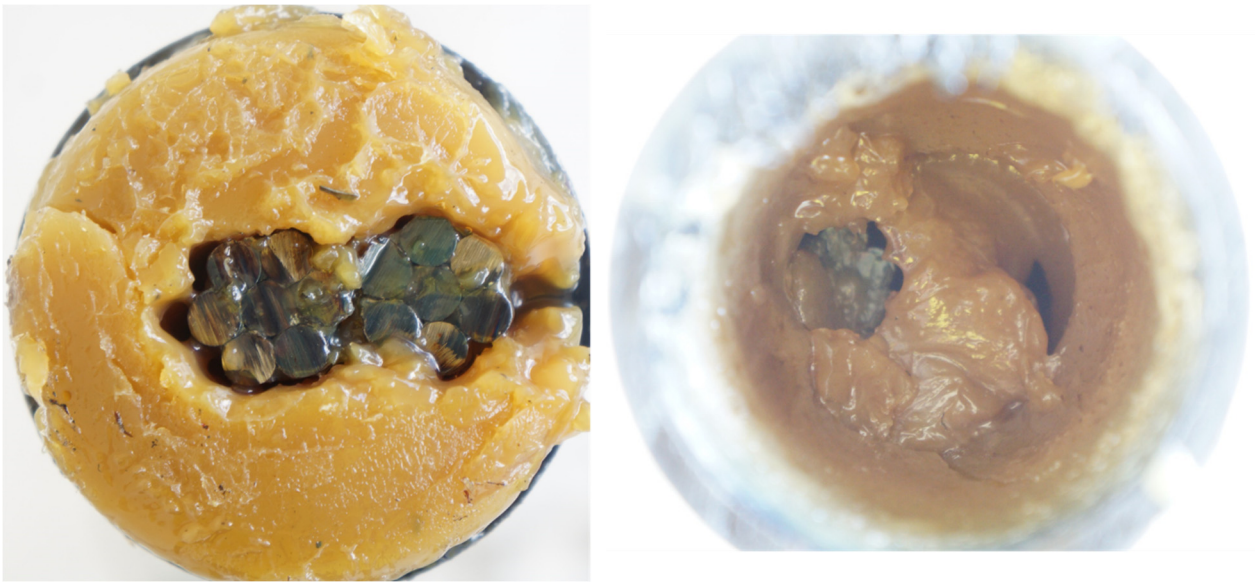


Figure 89. Civetea NC sample sprayed with fungi mix (one side) (June 2018).

Figure 90. Trenton NC sample sprayed with fungi mix (one side) (December 2017).

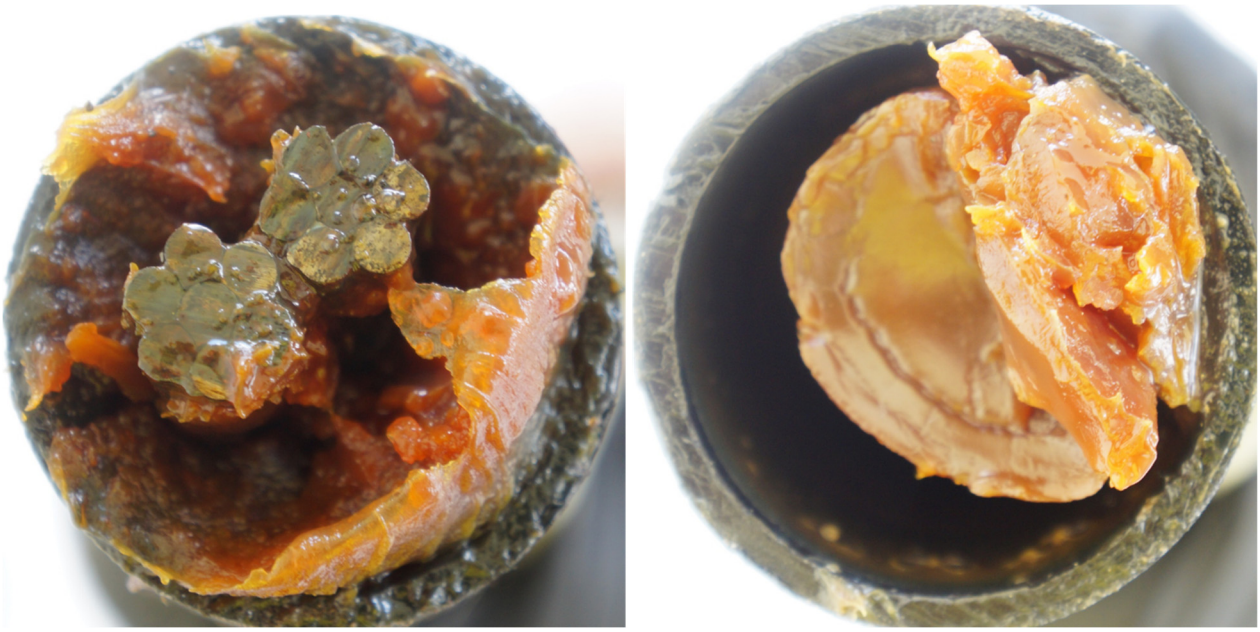


Figure 91. Trenton NC sample sprayed with fungi mix (one side) (June 2018).

Figure 92 shows the Sonneborn injected NC sample sprayed with fungi mix (December 2017). One of the strands shows corrosion on both ends, and not just the side sprayed (picture on the right). The corroding sites appear small. Figure 93 shows similar picture for the same sample during the visual inspection performed June 2018. The picture on the left now shows that the two strands have a thick flexible filler layer. The two pictures show that the filler has shifted positions on both sides of the sample. Figure 94 shows the Sanchem injected NC sample sprayed with fungi mix. No strand is visible on the side sprayed with the fungi mix due to a thick layer of the filler. The strands visible on the opposite side (picture on the left) do not show corrosion. Figure 95 shows the view observed during June 2018. No significant change occurred, except for a thicker filler layer covering the strands on the left. The change in the color of the filler was due to the use of flash in June 2018.

Figure 92. Sonneborn NC sample sprayed with fungi mix (one side) (December 2017).

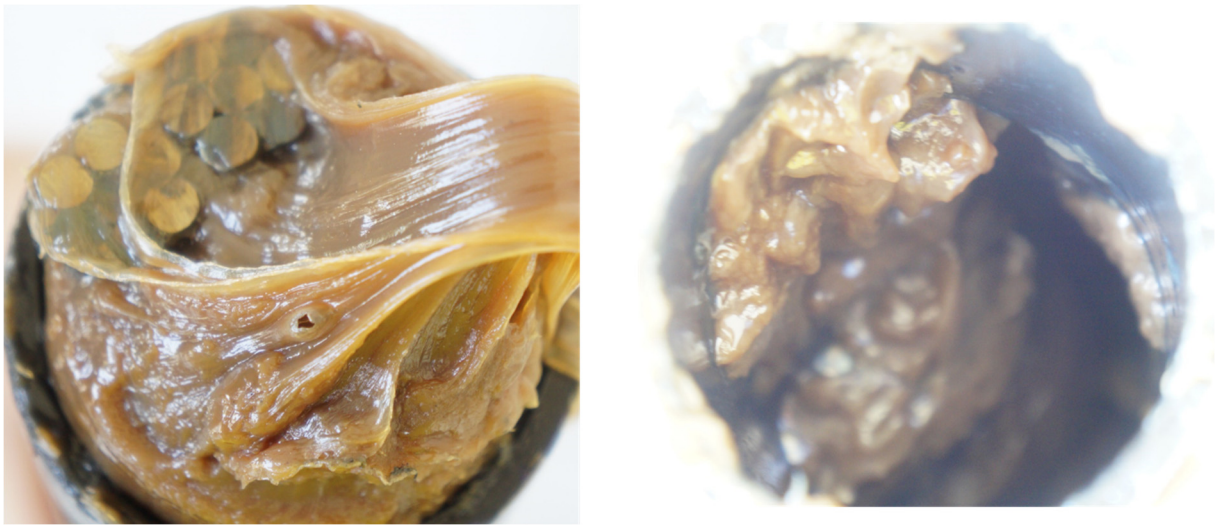


Figure 93. Sonneborn NC sample sprayed with fungi mix (one side) (June 2018).

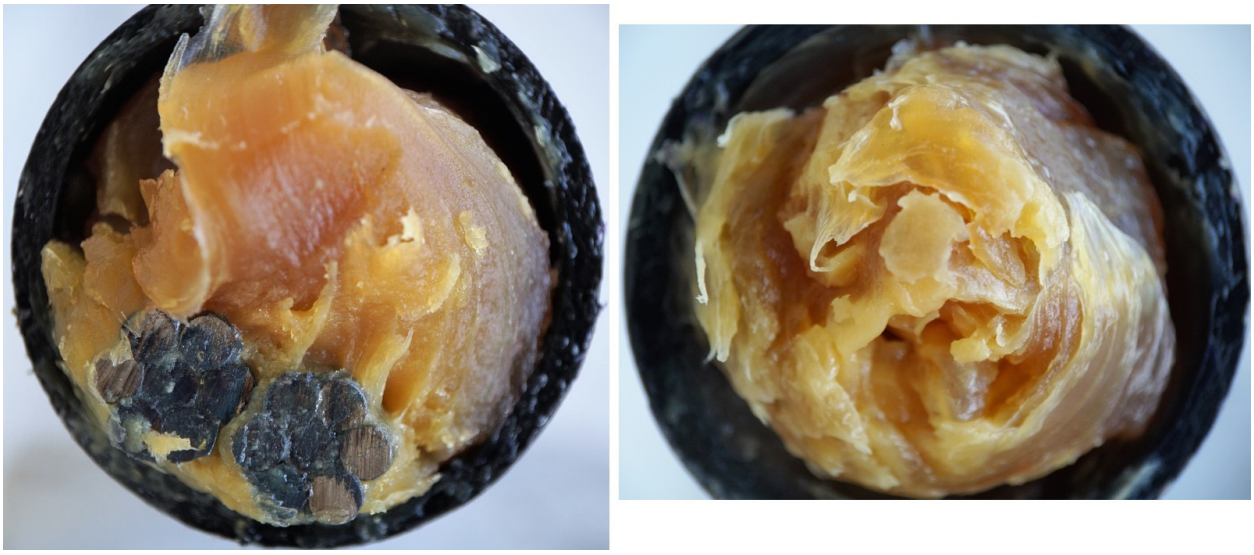


Figure 94. Sanchem NC sample sprayed with fungi mix (one side) (December 2017).

Figure 95. Sanchem NC sample sprayed with fungi mix (one side) (June 2018).

Similarly, Figure 96 shows pictures taken in December 2017 for the Nontribos injected NC sample that was sprayed with fungi mix. The picture on the right shows the side that was sprayed with fungi mix and no strand is visible. It is interesting to note that the filler shown in the picture on the right appears to have reacted at the surface (different color and texture). Figure 97 shows pictures taken in June 2018 for the samples injected with Nontribos and sprayed with fungi mix. The picture on the left shows that the strands are not corroding, and that additional filler has flowed down. The change in color seen is due to the use of flash for the pictures taken in June 2018.

Figure 96. Nontribos NC sample sprayed with fungi mix (December 2017).

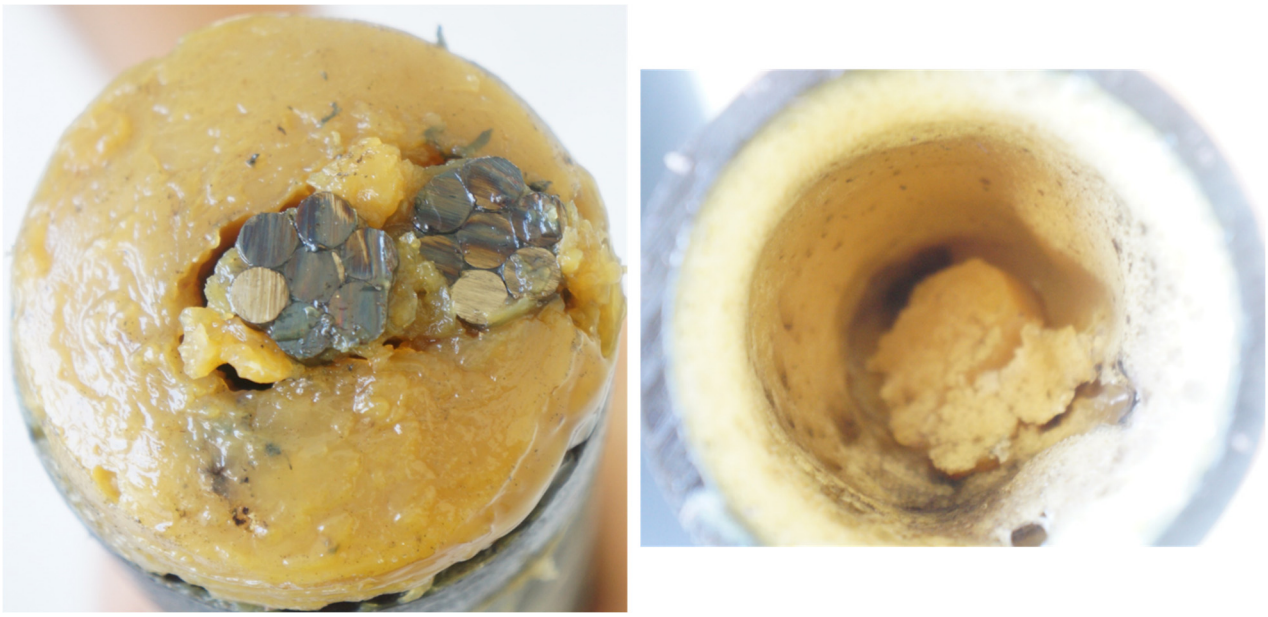


Figure 97. Nontribos NC sample sprayed with fungi mix (June 2018).

Figure 98 and Figure 100 show pictures taken in December 2017 of the overheated, injected samples for Trenton and Civetea, respectively. No strands were included in these samples. The sprayed side appears to have experienced a color change, but similar color change occurred on the sample sprayed with water. The color change might be due to overheating, and not a result of spraying with fungi mix solution. This applies to the pictures taken during December 2017. In June 2018, Figure 99 shows in the picture on the right that Trenton sample sprayed with fungi mix has a gold color stain on half of the surface that might be due to fungal interaction with the overheated Trenton filler. No such change was observed on the Civetea sample (Figure 101) during the inspection performed in June 2018.

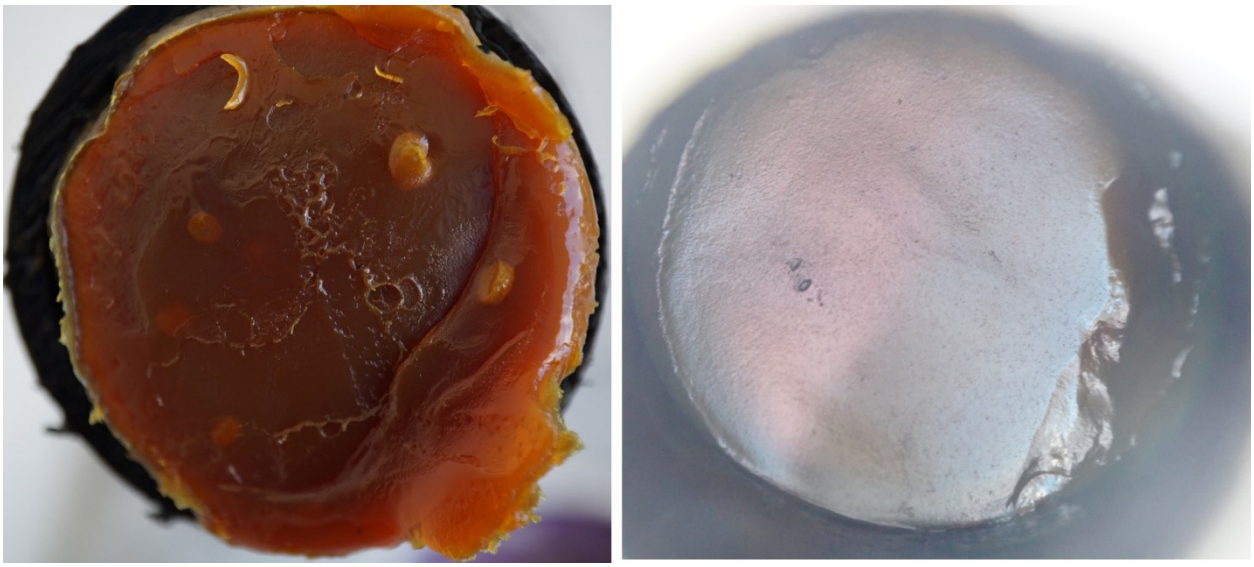


Figure 98. Trenton overheated sample sprayed with fungi mix (one side) (December 2017).

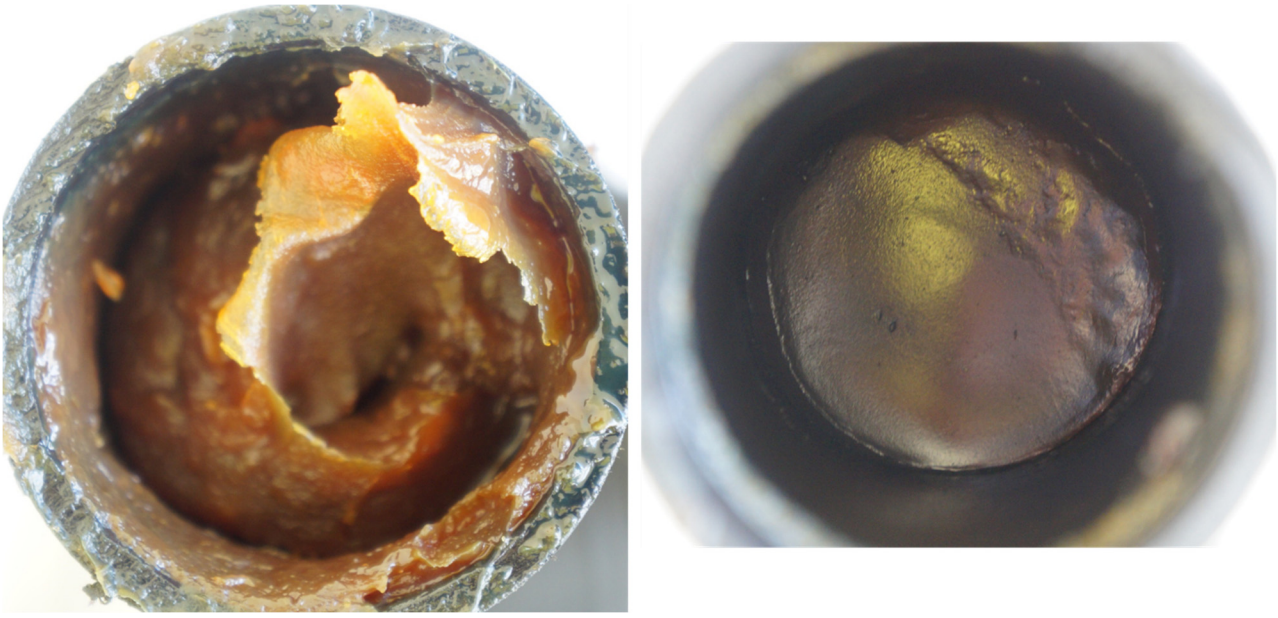


Figure 99. Trenton overheated sample sprayed with fungi Mix (one side) (June 2018).

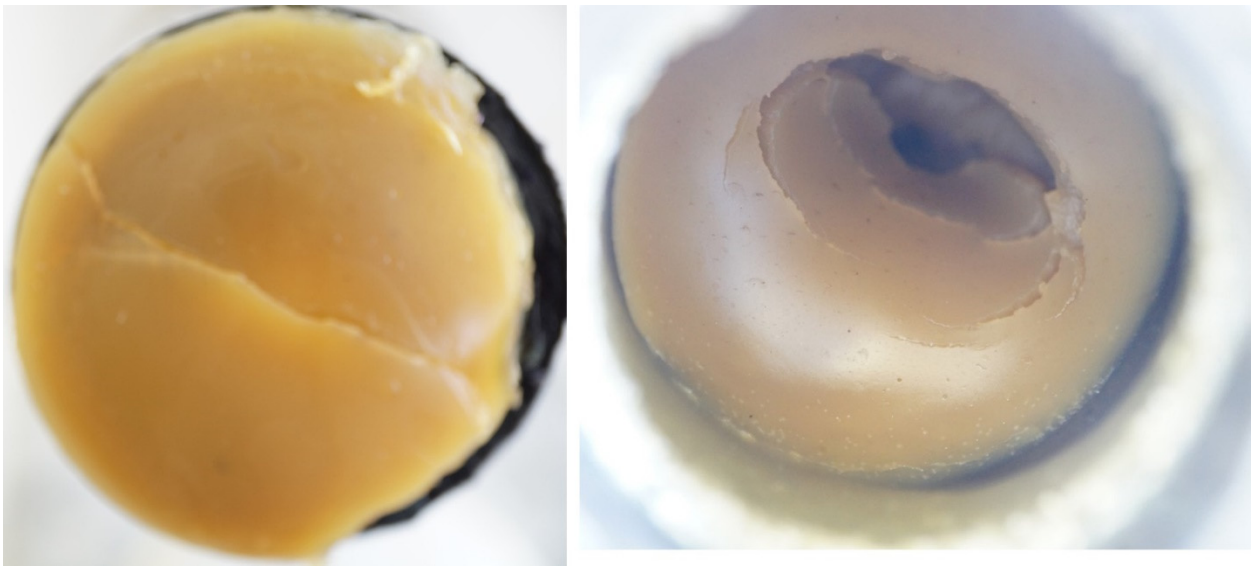


Figure 100. Civetea overheated sample sprayed with fungi mix (one side) (December 2017).

Figure 101. Civetea overheated sample sprayed with fungi mix (one side) (June 2018).

4.1.3 Two-inch-diameter – 4-foot-long Polycarbonate Samples Injected with Various Fillers Placed at the Partially Sheltered (2nd floor) Location.

Pictures of the samples sprayed with water and pictures of the samples sprayed with fungi mix will be presented in this section. In the figures that follow, the picture on the right shows the side that was sprayed. The samples that used NC strands were selected. A third sample with strands subjected to Conditioning Type 1 (C1) were placed on the shelf at this exposure location, but were not sprayed. The caps of C1 samples were periodically removed, similar to what was done for NC sprayed samples. No changes were observed on the C1 samples, because neither moisture nor fungi mix solution was sprayed. Figure 102 shows the NC samples sprayed with fungi mix, for specimens injected with Civetea, Nontribos, and Sanchem for the inspection performed in December 2017. Figure 103 shows NC samples sprayed with fungi mix, for specimen injected with Civetea, Nontribos, and Sanchem for the inspection done in May 2018. The samples on the right show modest amounts of corrosion on the samples injected with Civetea and Nontribos, but not on the sample injected with Sanchem. The opposite side did not show corrosion on any of these samples. The corrosion appears to have progressed on the Civetea sample. Also a change in the coloring of the flexible filler took place. A lesser amount of drops/moisture were observed during May 2018.

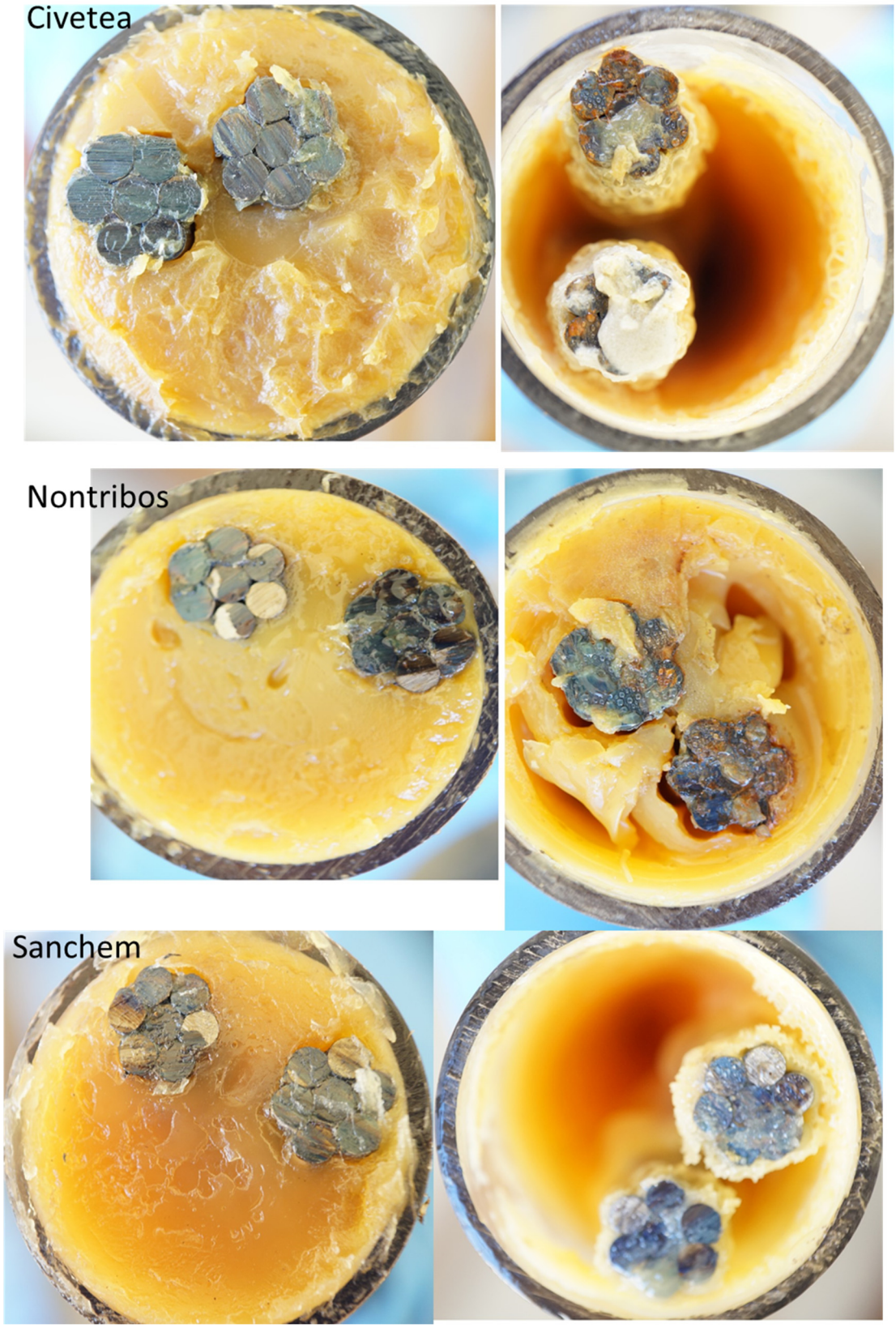


Figure 102. Samples injected with flexible fillers and sprayed with fungi mix, (December 2017)

Figure 103. Samples injected with flexible fillers (polycarbonate tubing) and sprayed with fungi mix (May 2018).

Figure 104 show pictures taken in December 2017 for samples injected with Trenton and Sonneborn fillers, and sprayed with fungi mix. The sample injected with Sonneborn does not show

corrosion, whereas a few wires of one strand of the sample injected with Trenton show a modest amount of corrosion. Figure 105 shows pictures taken in May 2018 for Trenton and Sonneborn injected samples that were sprayed with fungi. No significant changes took place on the Sonneborn sample. The picture on the right shows red rust like coloring on one of the strands coated with Trenton.

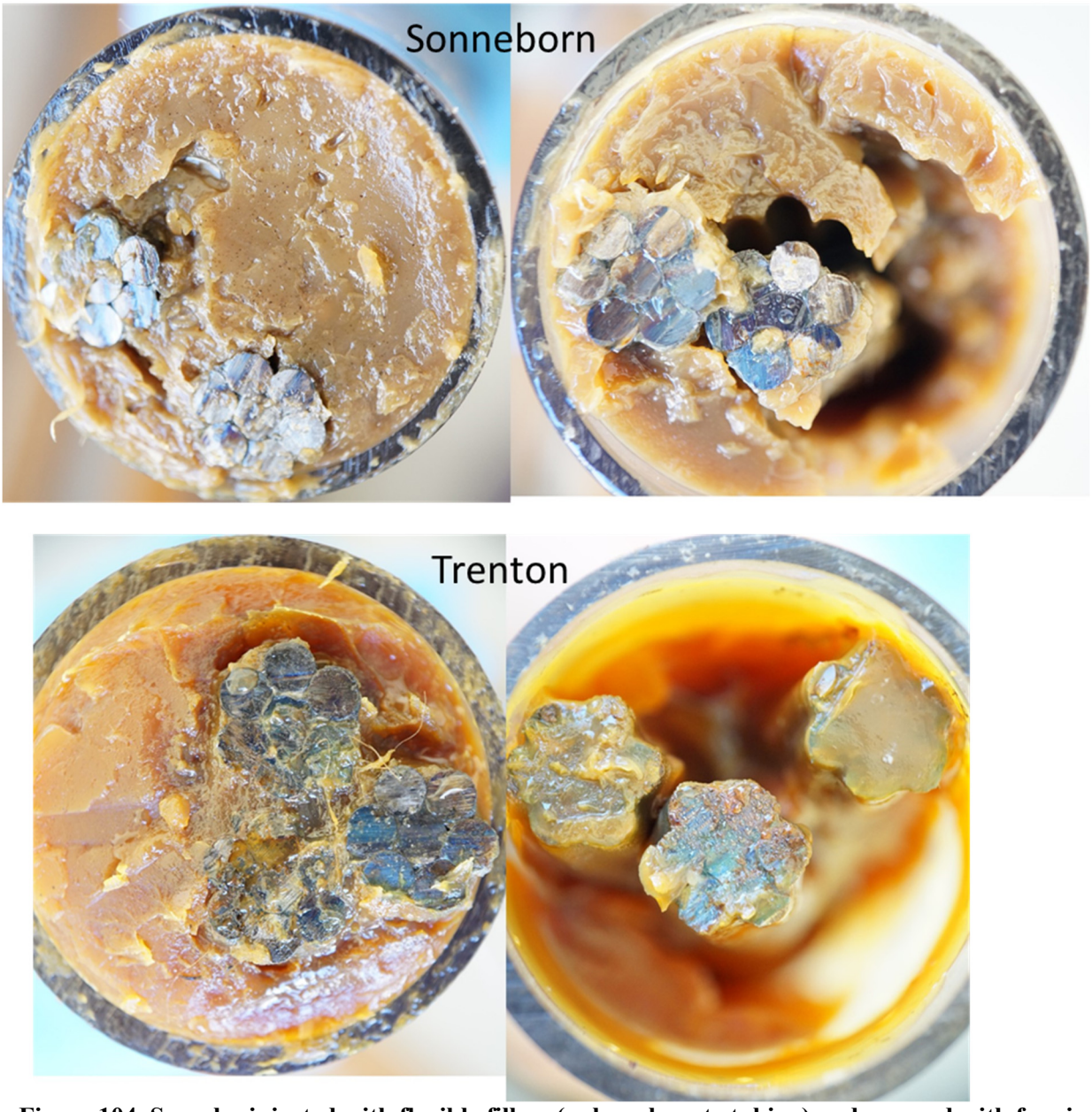


Figure 104. Samples injected with flexible fillers (polycarbonate tubing) and sprayed with fungi mix, (December 2017)

Figure 105. Samples injected with flexible fillers (polycarbonate tubing) and sprayed with fungi mix, (May 2018)

Figure 106 shows the surface condition for samples sprayed with water and injected with Civetea, Nontribos and Sanchem during the December 2017 inspection. Minor amounts of rust are visible on one strand of the sample injected with Civetea (top right picture). A couple of wires on one strand show corrosion spots on the sample injected with Nontribos, but no corrosion spots are distinguishable on the sample injected with Sanchem. Figure 107 shows pictures taken in May 2018 of samples sprayed with water and injected with Civetea, Nonbribos and Sanchem. The corrosion spots appear to be at the same locations, and at some there is a thicker red rust-like coloring. Figure 108 shows pictures of the surface condition for specimens injected with Sonneborn and Trenton, and sprayed with water during December 2017. No corrosion is visible on these samples. Figure 109 shows pictures of the surface condition for specimens injected with Sonneborn and Trenton, and sprayed with water during May 2018. No corrosion is visible on wires

of the strands coated with Sonneborn. The picture on the right shows some red rust-like coloring on the wires of one of the strands for the sample injected with Trenton and sprayed with water.

Figure 106. Samples injected with flexible fillers (polycarbonate tubing) and sprayed with water. (December 2017)

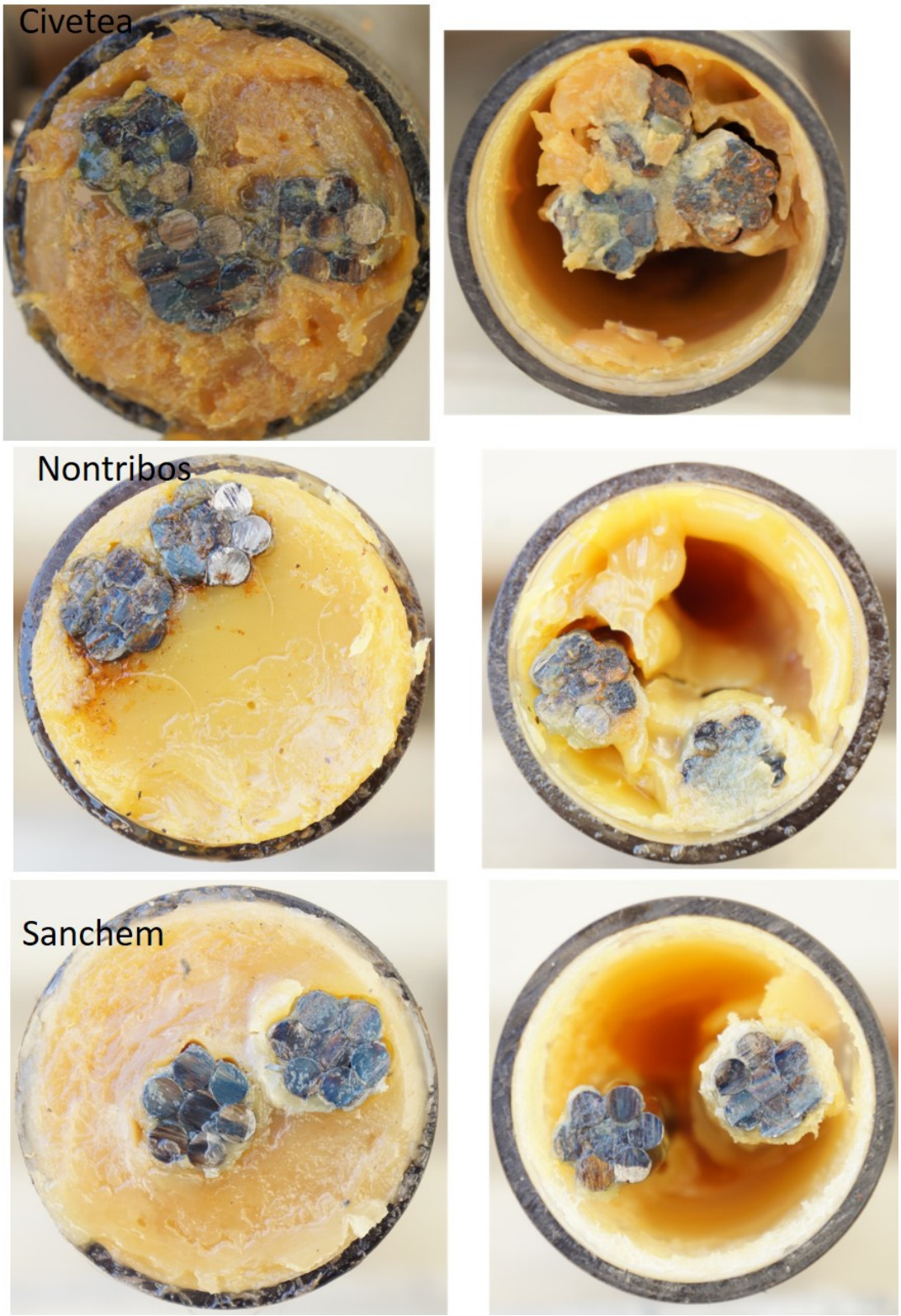


Figure 107. Samples injected with flex fillers (polycarbonate tubing) and sprayed with water (May 2018).

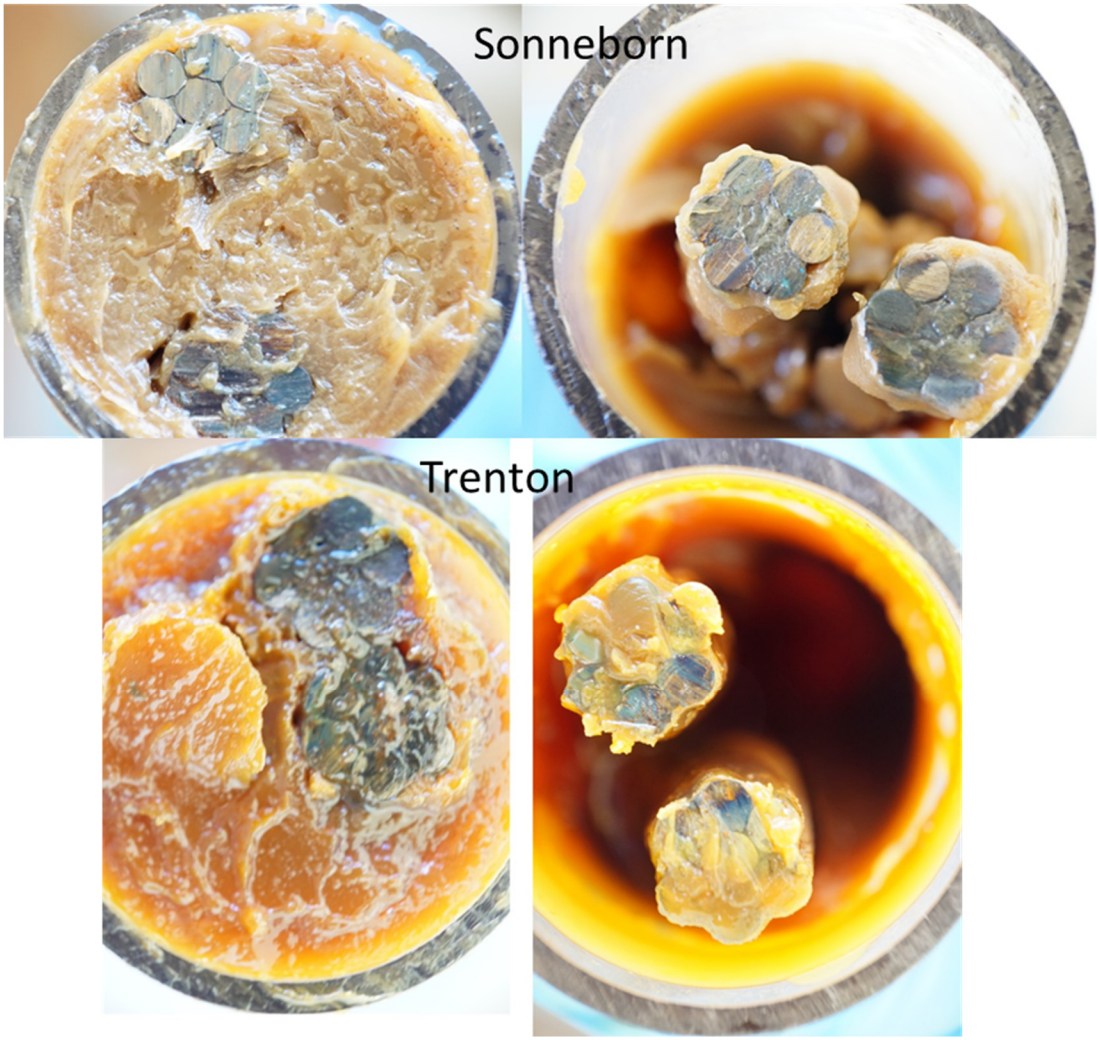


Figure 108 Samples injected with flexible fillers (polycarbonate tubing) and sprayed with water. (December 2017)

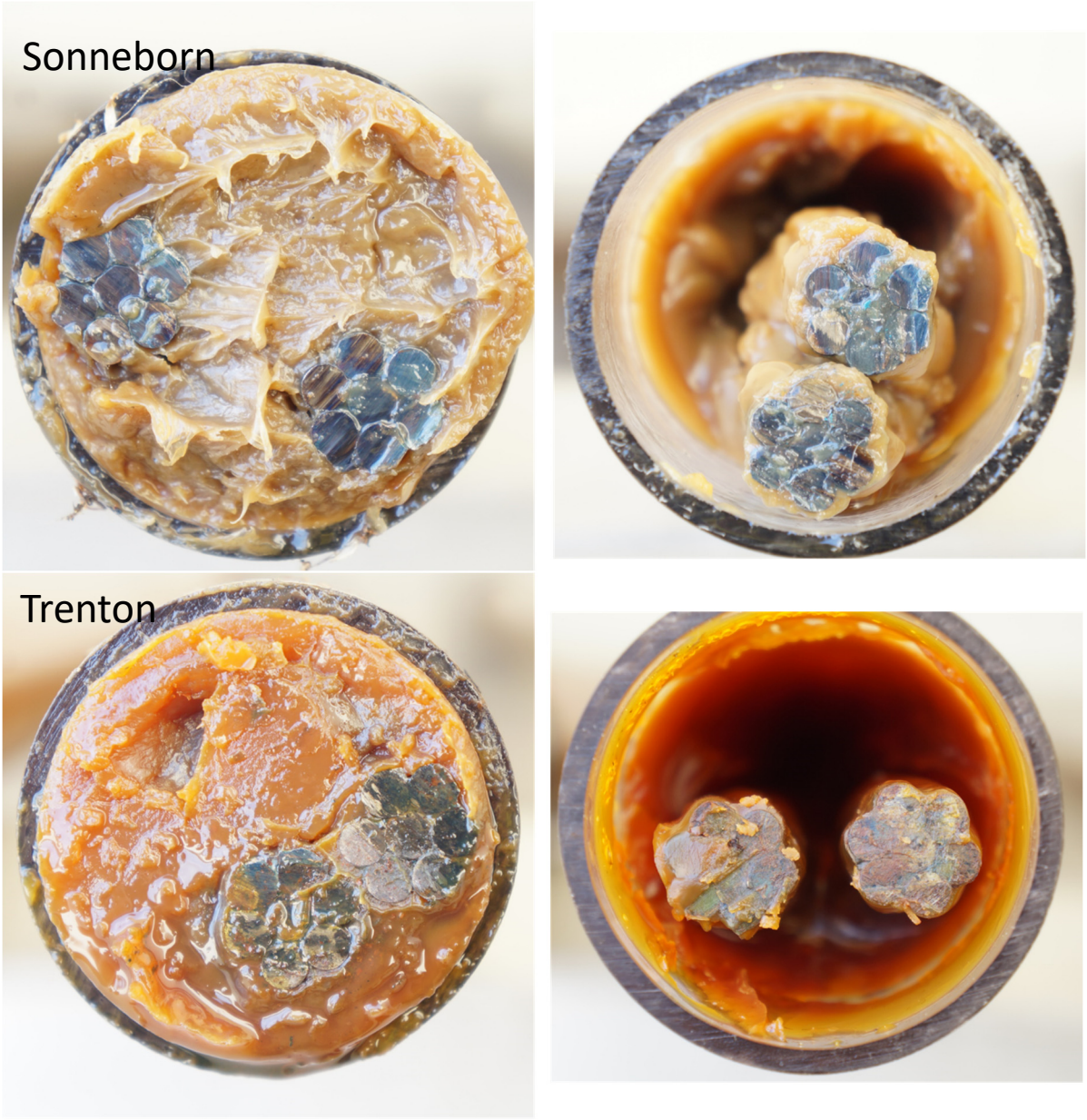


Figure 109. Samples injected with flexible fillers (polycarbonate tubing) and sprayed with water (May 2018).

4.1.4 Laboratory Exposure: 2-foot-long Samples

This section describes the condition of the 2 foot long samples injected with neat fillers. The samples contained two or three strands. These samples had one side of the sample sprayed with fungi mix.

Figure 110 shows both ends of the sample injected with Civetea (December 2017). The picture on the right shows the side sprayed with fungi mix. No corrosion sites appear on the picture on the right, but both strands show wires with rust on end that was not sprayed(picture on the left). From the picture it appears that part of the strand surface appears not to have a filler layer and rust could be due to atmospheric corrosion while exposed in the lab, and also could be due to periodic

exposure to the lab environment while inspecting the sample. Figure 111 shows the same sample during the inspection performed in June 2018. No corrosion was observed on the side sprayed with fungi mix (the picture on the right). The picture on the left shows larger coverage by corrosion products (red rust). It is possible that some of the solution wicked with time and reached the strand that has little or no flexible filler. Figure 112 shows both ends of the samples injected with Trenton and sprayed with fungi mix, during the inspection that took place in December 2017. No corrosion is observed. The side sprayed with fungi mix appears to have a layer of the filler. Figure 113 shows in the picture on the right that both strands have a few wires with red rust-like stains.

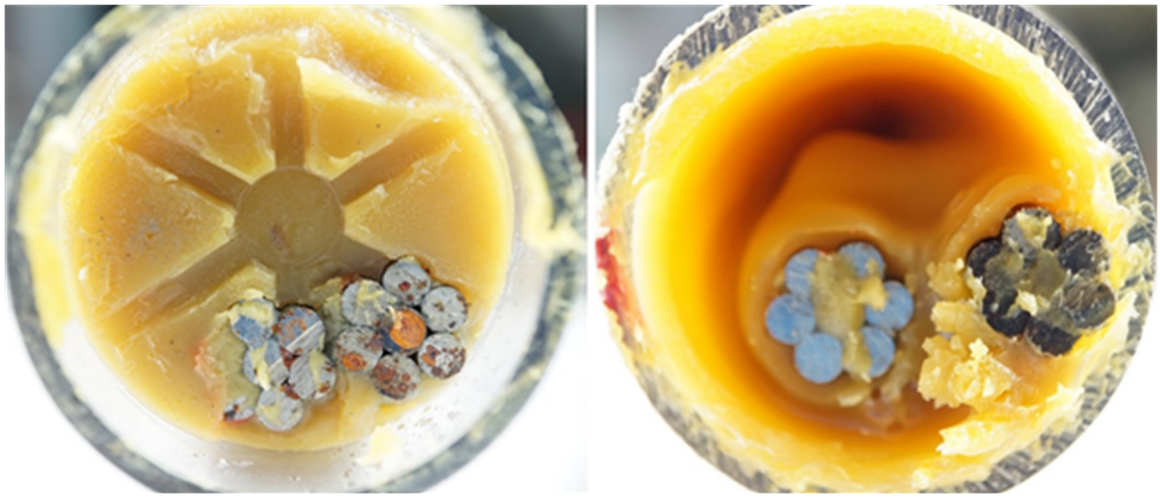


Figure 110. Two-foot long samples injected with Civetea filler (polycarbonate tubing) sprayed with fungi mix (December 2017).

Figure 111. Two-foot long samples injected with Civetea filler (polycarbonate tubing) sprayed with fungi mix (June 2018).

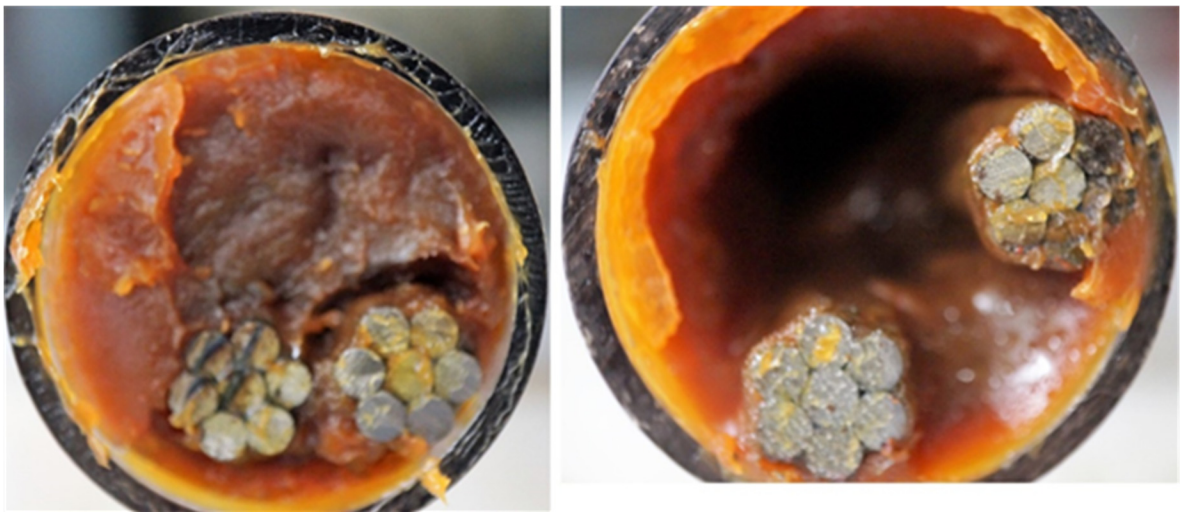


Figure 112. Two-foot long samples injected with Trenton filler (polycarbonate tubing) sprayed with fungi mix (December 2017)

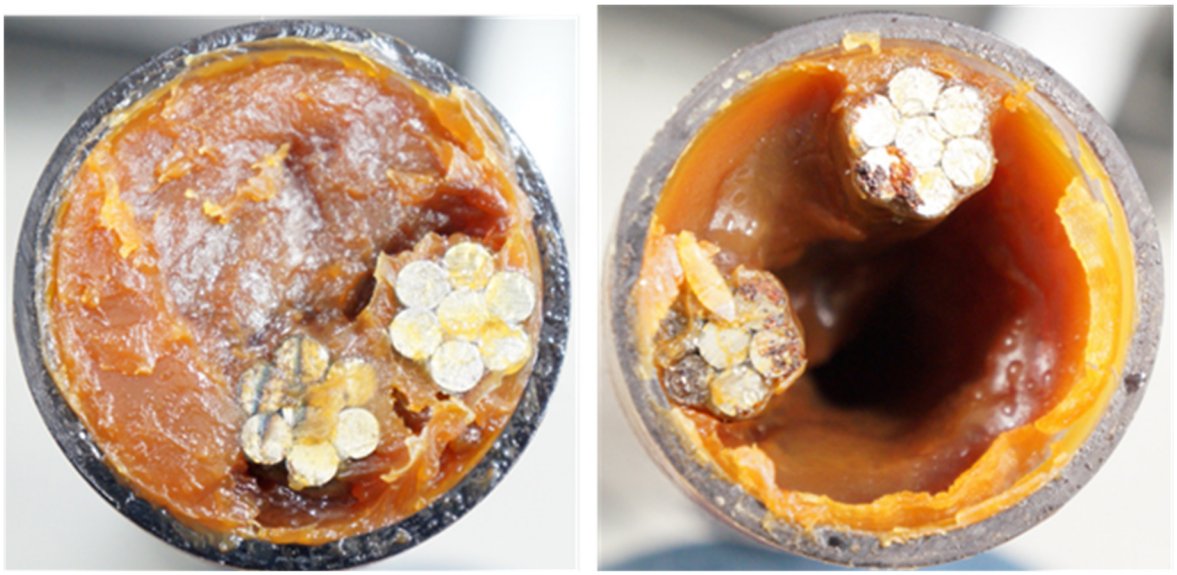


Figure 113. Two-foot long samples injected with Trenton filler (polycarbonate tubing) sprayed with fungi mix (June 2018).

Figure 114 shows the samples injected with Sonneborn and sprayed with fungi mix. The picture on the right (the side sprayed with fungi) illustrates that only one strand is visible, and that the other is fully covered with filler. No corrosion is observed. The picture on the left shows no corrosion on the wires. Figure 115 shows pictures taken in June 2018 for the sample injected with Sonneborn and sprayed with fungi mix. No corrosion is visible. The recent pictures were taken with the flash on and that caused the filler to look lighter and some of the wires to appear quite bright.

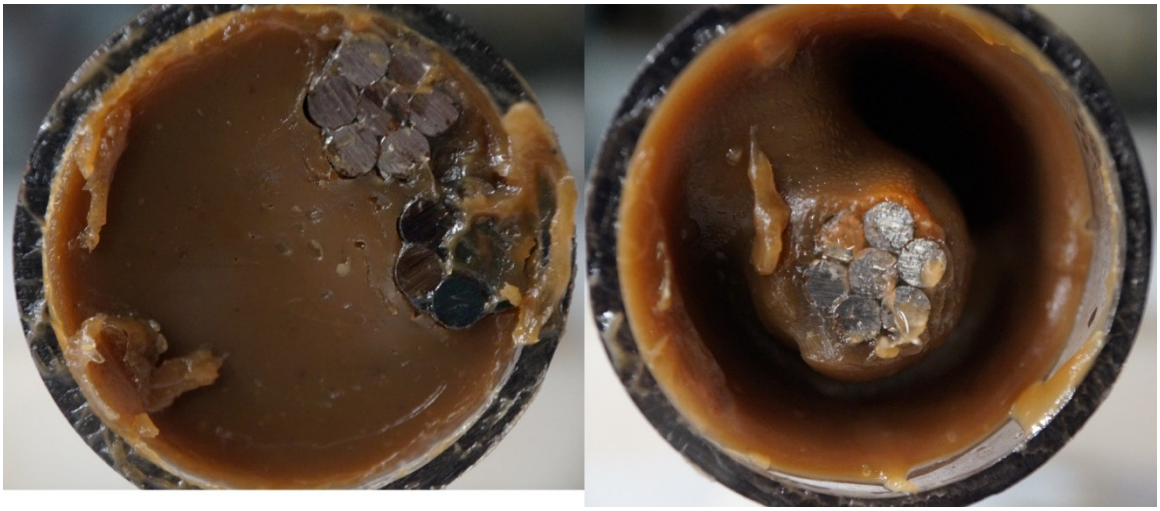


Figure 114. Two-foot long samples injected with Sonneborn filler (polycarbonate tubing) sprayed with fungi mix ((December 2017)

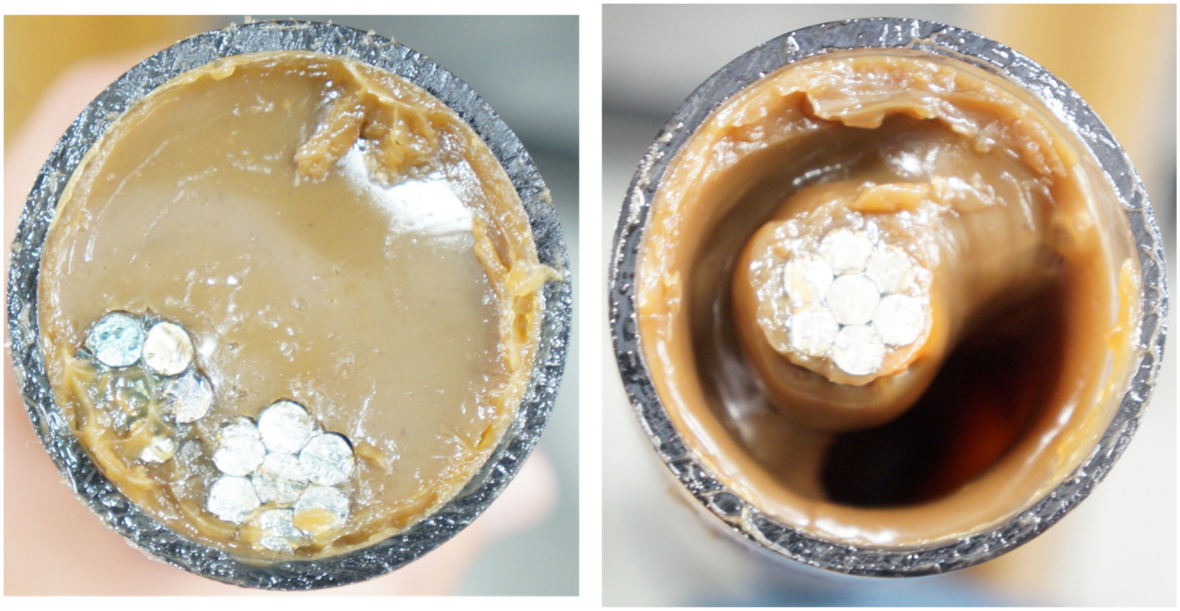


Figure 115. Two-foot long samples injected with Sonneborn filler (polycarbonate tubing) sprayed with fungi mix (June 2018).

Figure 116 shows the sample injected with Sanchem sprayed with fungi mix during the inspection that took place in December 2017. In the picture on the right (the side sprayed with fungi mix), both strands are visible and both appear to be covered with filler. No corrosion is observed. The picture on the left shows black spots. Figure 117 shows pictures taken in June 2018 on the same sample. Flash was used and resulted in a lighter coloring of the flexible filler in the picture. The picture on the left shows that most of the black stains observed in December 2017 are no longer visible, but there are still some areas covered in black. The picture on the right shows that two wires have reddish stains.

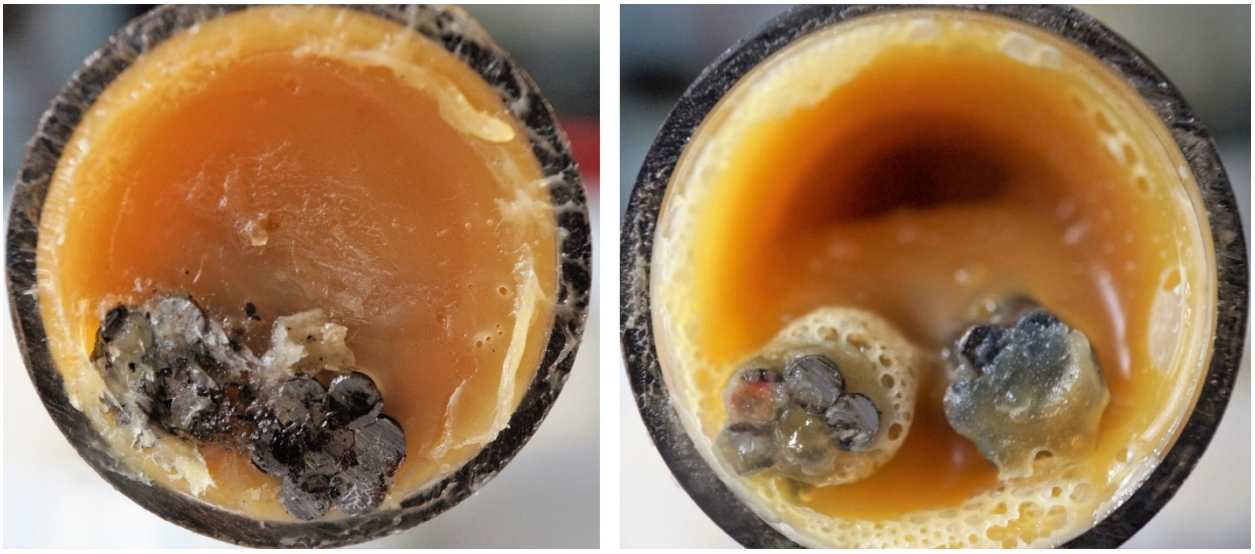


Figure 116. Two-foot long samples injected with Sanchem filler (polycarbonate tubing) sprayed with fungi mix (December 2017)

Figure 117. Two-foot long samples injected with Sanchem filler (polycarbonate tubing) sprayed with fungi mix (June 2018).

Finally, Figure 118 shows the sample injected with Nontribos and sprayed with fungi mix during the visual inspection that took place in December 2017. The side sprayed with fungi (picture on the right) shows good coverage of the filler over the strands. The picture on the left shows a couple of rust-colored spots on a couple of wires, but most other wires appear to be covered with the filler. Figure 119 shows the pictures taken in June 2018 on the Nontribos Sample Sprayed with fungi mix. The picture on the right shows that all wires still have good layer of the filler, but on the side there might be a couple of spots showing red rust stains. The figure on the left shows that corrosion has progressed on the wires of the strand shown on the left.

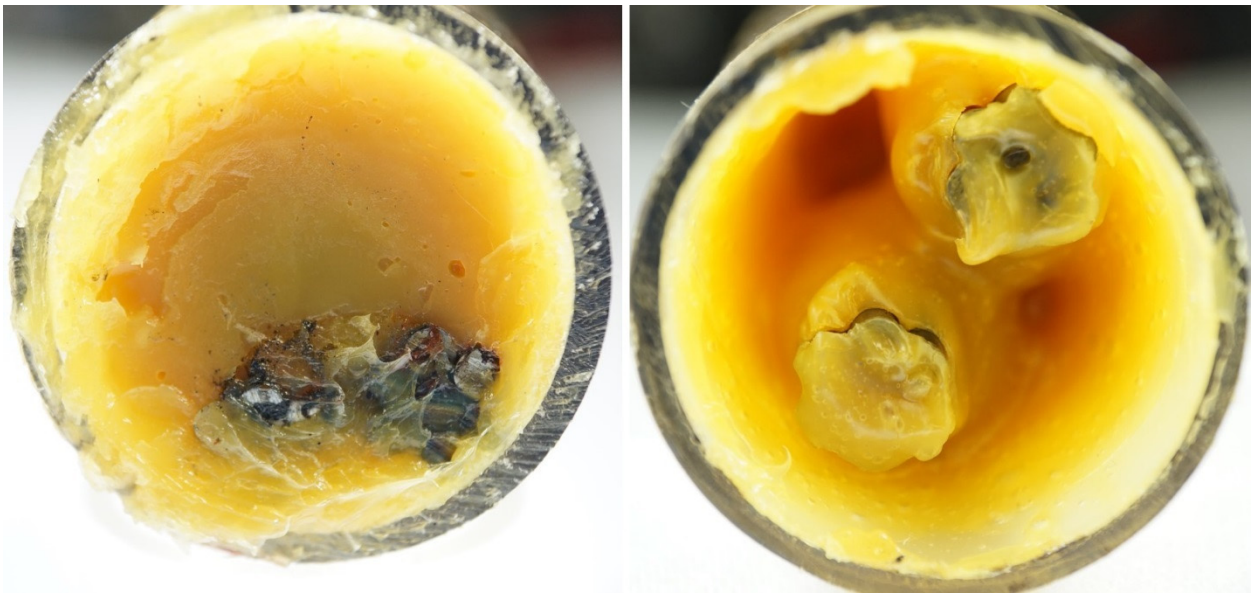


Figure 118. Two-foot long samples injected with Nontribos filler (polycarbonate tubing) sprayed with fungi mix (December 2017)

Figure 119. Two-foot long samples injected with Nontribos filler (polycarbonate tubing) sprayed with fungi mix (June 2018)

4.2 Summary

The visual inspection of the four inch diameter samples injected with Trenton and sprayed with a variety of solutions (water, fungi mix, each individual fungi once then water, and no sprayed) indicate that either there is no corrosion or there is very minor corrosion that is on-going (even during the inspection that took place in June 2018). It appears that if the samples are exposed to sporadic moisture (with or without fungi), the water evaporates and no corrosion or minor corrosion occurs. Recall that the solution sprayed each time was less than 1.5 milliliters, larger amounts might produce a different result.

Two inch diameter samples with filler injected in HDPE tubing and sprayed with fungi mix did not show corrosion on the side sprayed with fungi.

Some of the two inch diameter four foot long samples with filler injected into polycarbonate tubing and sprayed with fungi mix showed corrosion. A thin filler coating or no coating at all likely contributed to corrosion initiating. The samples on the right (sprayed side) on most figures shows modest amount of corrosion on the samples injected with Civetea, Trenton, and Nontribos, but not on the samples injected with Sanchem or Sonneborn. For samples sprayed with water, only the sample injected with Civetea showed some corrosion spots on one of the strands.

Two inch diameter two foot long samples with filler injected into polycarbonate tubing and sprayed with fungi mix while exposed in the lab environment did not show much corrosion on the side sprayed with fungi. In a couple of samples, rust spots were observed on the side that was not sprayed (possibly due to solution wicking). The corrosion spots were more pronounced on the samples in pictures taken during the more recent inspection (June 2018)

Chapter 5 - Forensic Analysis

Visual inspection was performed on filler-coated samples (some sprayed with water, others with fungi mix). Coated strands were provided by SRC. The samples were exposed at the semi–sheltered location on April 03, 2017. The solution was first applied on May 5, 2017. The samples were stored inside PVC segments.

The following is the timeline of when corrosion was first observed. On September 19, 2017, two out of the three samples sprayed with fungi mix presented rust (Trenton and Nontribos), On September 19, 2017 the Nontribos sample sprayed with water and the Nontribos not sprayed sample presented rust. By October 3, 2017 the corresponding solution was sprayed on the samples. On October 25, 2017, the strand coated with Civetea presented a few rust spots on the sample sprayed with fungi mix and the sample sprayed with water. The coated strands were periodically removed from the casing. The PVC ends have PVC caps but these are not glued. Pictures were taken on a few instances. After the inspection in late February 2018, several samples were selected to have a section cut off for forensic examination. Selected samples were cut after 280 days of exposure and the remaining segments continue to be exposed.

Sections 10 to 12 centimeter long were cut off from each of the coated strands sprayed with fungi. Sections were also cut off from samples sprayed with water and coated with Nontribos and Civetea, but not from the strand coated with Trenton. The filler was manually removed and then mineral spirits were used to remove any remaining filler. Then the wires showing the most corrosion were selected for chemical cleaning using ASTM G1. The following pages document the visual inspection. In some cases, a figure showing a picture of the seven wires precedes the slide with pictures of close-ups for locations that showed the most corrosion extent on selected wires.

This section describes the visual inspection done on the flexible filler-coated strands exposed at the semi-sheltered location and that were sprayed with fungi mix or with water.

In general the wires from samples that were sprayed with fungi mix appear to show the most corrosion when compared to those sprayed with water. The corrosion extended not just to the sites visible with the filler, but also beyond these sites. The wires coated with Nontribos appear to have the deeper corrosion sites. Superficial corrosion sites were observed on samples sprayed with fungi mix and coated with Trenton and Civetea. The extent of corrosion was significantly smaller on the samples cleaned that had been sprayed with water.

5.1 Trenton-coated Strand Sprayed with fungi mix

Figure 120 shows after cleaning wires coated with Trenton. This picture shows six of the seven wires. At least four of the wires were chemically cleaned. Figure 121 shows selected close-ups of the locations that appear to have the worst corrosion. One of these wire sections was selected for further analysis by taking images with the SEM. A smaller section that fits within the SEM holder was obtained. Figure 122 shows the images obtained with the SEM. The image taken with 26X magnification suggests that corrosion took place at various locations. The location close to the center was selected for images at 150X, 300X, and 1,000X. The pictures obtained at 150X and 300X shows that on the left side of the images there are striations and on the center there is a region that had more depth (indicating larger cross-section loss).

Figure 120. Section of Trenton coated strand (fungi mix), after cleaning

Figure 121. Close-up selected wires, after cleaning. (Trenton – fungi mix)

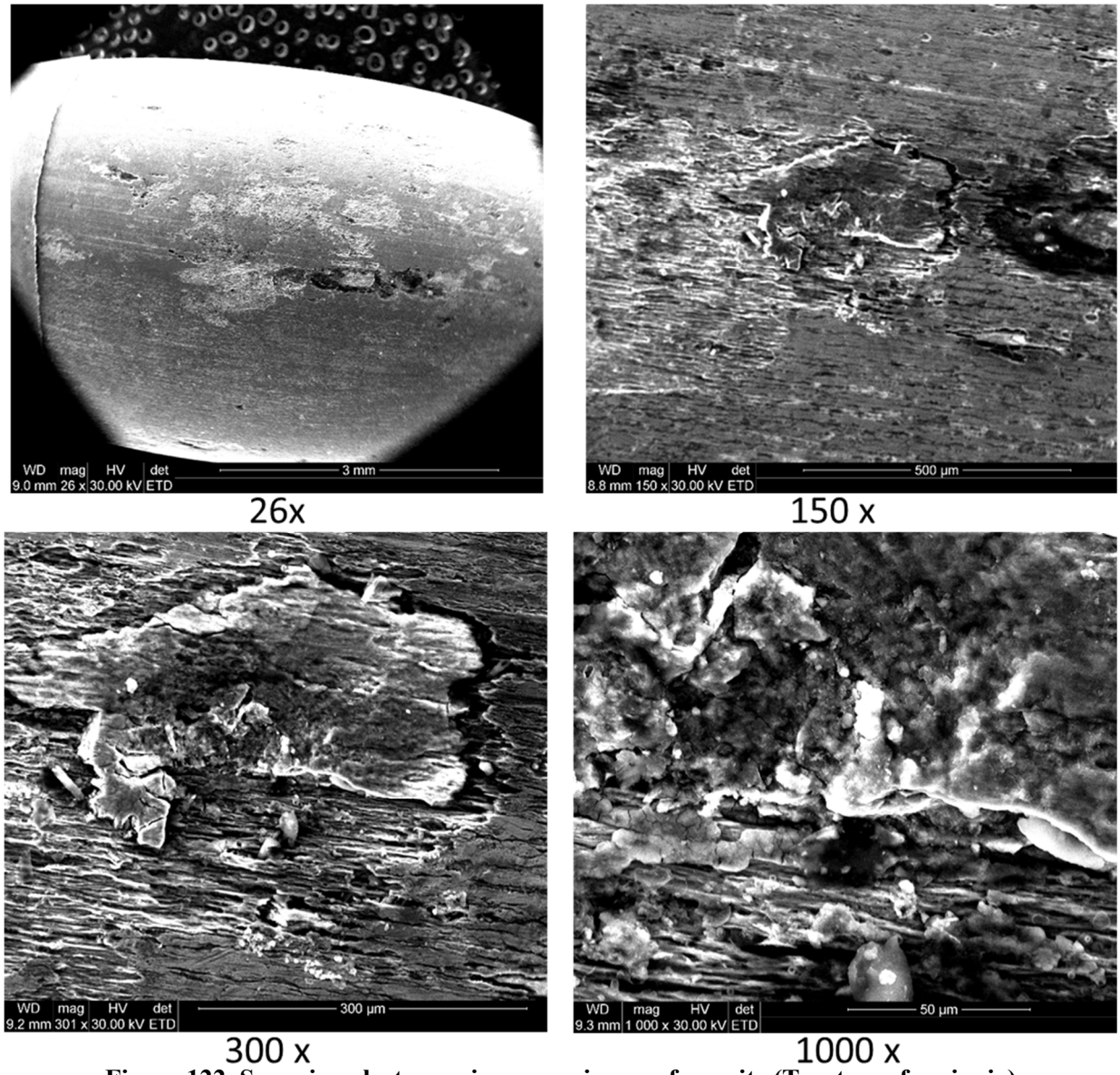


Figure 122. Scanning electron microscope images for a site (Trenton – fungi mix)

5.2 Civetea-coated Strand Sprayed with fungi mix

Figure 123 shows the wires after cleaning for the section of the strand that was cut off from the sample coated with Civetea and sprayed with fungi mix. All wire sections were cleaned with mineral spirits, but only four were subjected to chemical cleaning (bottom four). The center wire (straight wire) appears to have the larger number of corroding sites. Corrosion spots are observed on the other three wires cleaned. Figure 124 shows close-ups for three of the wires. Figure 125 shows a picture taken with a stereo microscope and five pictures taken with the SEM. The SEM images show striations at the different magnifications and are more easy to identify on images taken at ≥ 300X.

Figure 123. Section of Civetea coated strand, after cleaning (fungi mix).

Figure 124. Close-up selected wires, after cleaning (Civetea – fungi mix)

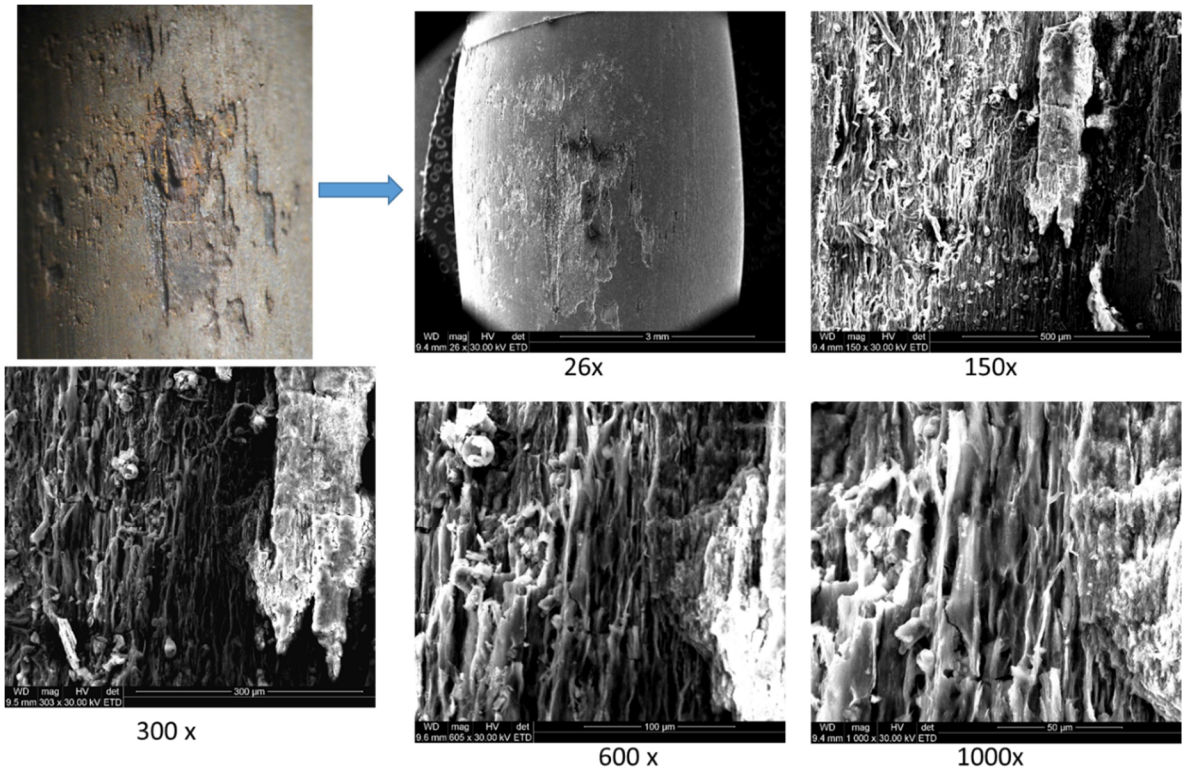


Figure 125. Scanning electron microscope images. (Civetea – fungi mix).

5.3 Nontribos-coated Strand Sprayed with fungi mix

Figure 126 shows a picture of four cleaned wires taken from the cut off strand section for the sample coated with Nontribos and sprayed with fungi mix. Three of the wires show corrosion spots. Figure 127 shows close-ups for three wires. The corrosion sites are more clearly seen in the close-up images. The images suggest minor cross-section took place on the spots where corrosion occurred. Two sections were cut off from one of the cleaned wires to be able to observe them via the scanning microscope. Figure 128 and Figure 129 show on each a stereo micrograph and five images taken with the SEM. Figure 128 shows images with the following magnifications: 26X, 54X, 150X, 1,000X, and 5,000X. The images with the three larger magnifications show striations pattern that are likely due to corrosion. The image taken at 5,000X shows needle like structures. Figure 129 shows a stereo microscope image and five images taken with the SEM. The regions chosen had a corrosion site with a different shape. Particles appear to be present at the deeper corrosion site and higher magnification (1,000X), there are regions that show striations at two different levels (150X and 300X images). Figure 130 shows that some regions of this wire had minor corrosion damage. Figure 131 shows a 2,000X magnification picture of a different site where the corrosion developed an interesting feature. The picture shows the remaning steel for a section that had corrosion cross-section loss.

Figure 126. Section of Nontribos coated strand, after cleaning (Nontribos - fungi mix)

Figure 127. Close-up selected wires, after cleaning (Nontribos fungi mix)

)

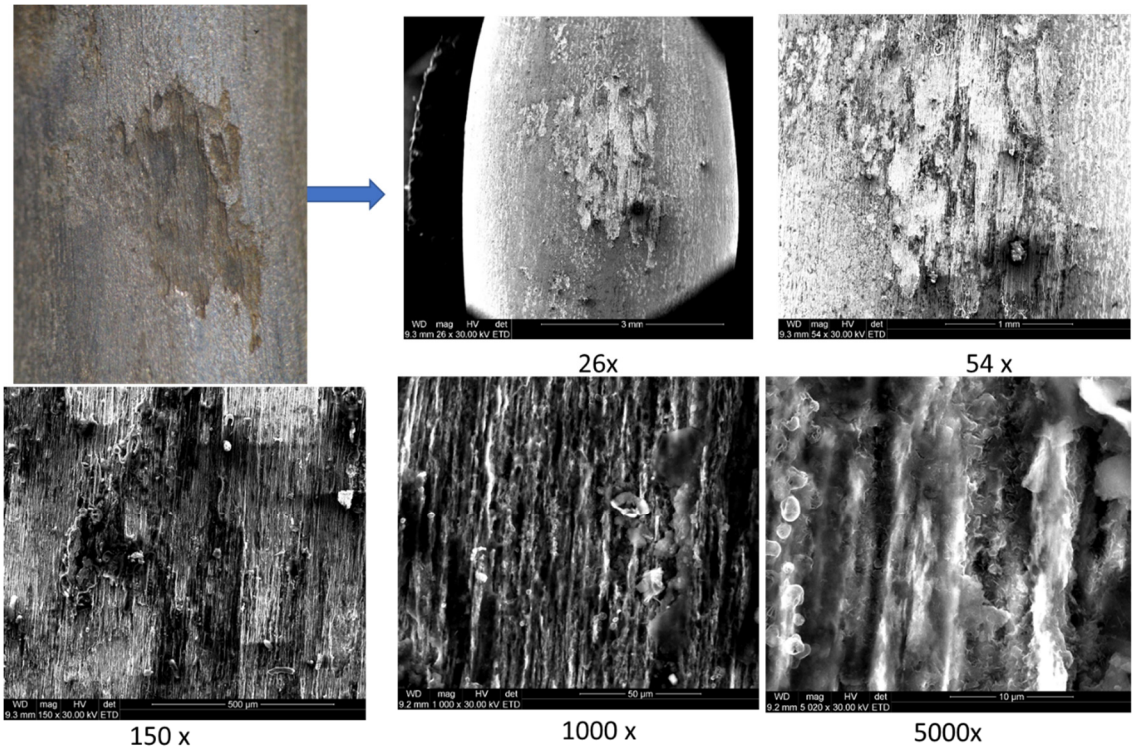


Figure 128. Scanning electron microscope images for site 1 (Nontribos - fungi mix)

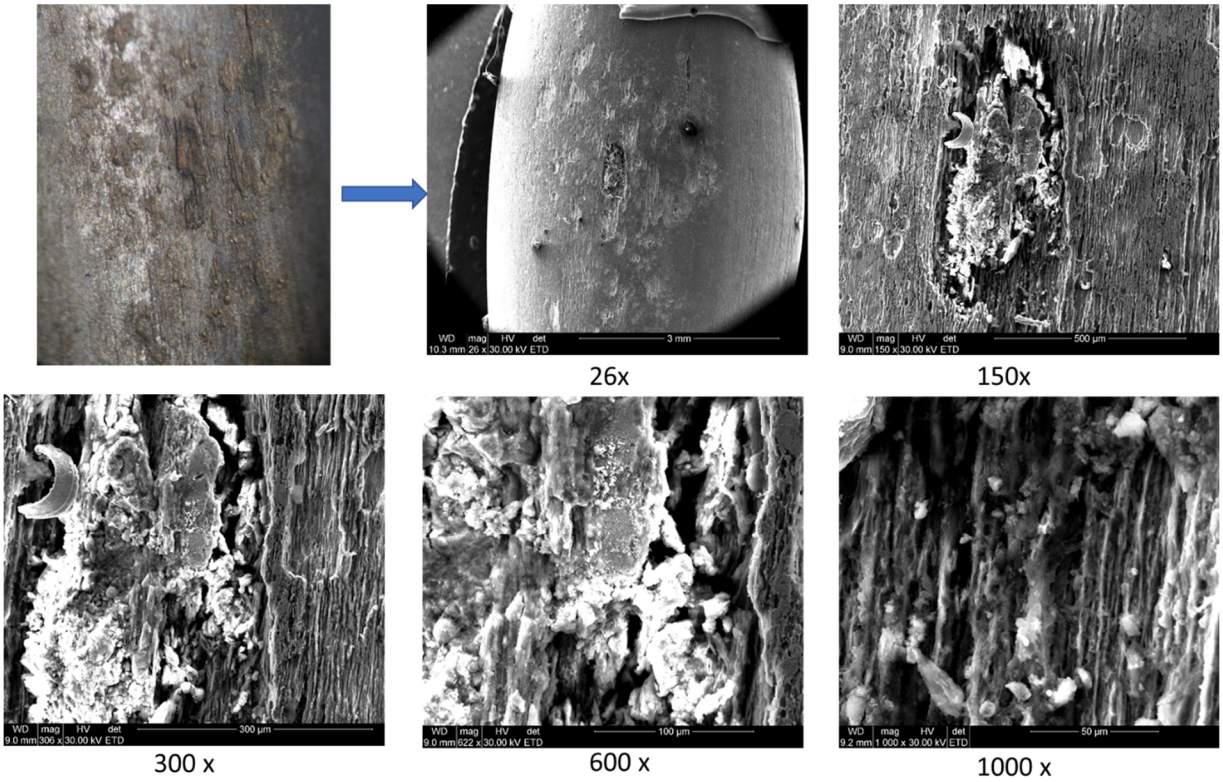


Figure 129. Scanning electron microscope images for site 2 (Nontribos - fungi mix)

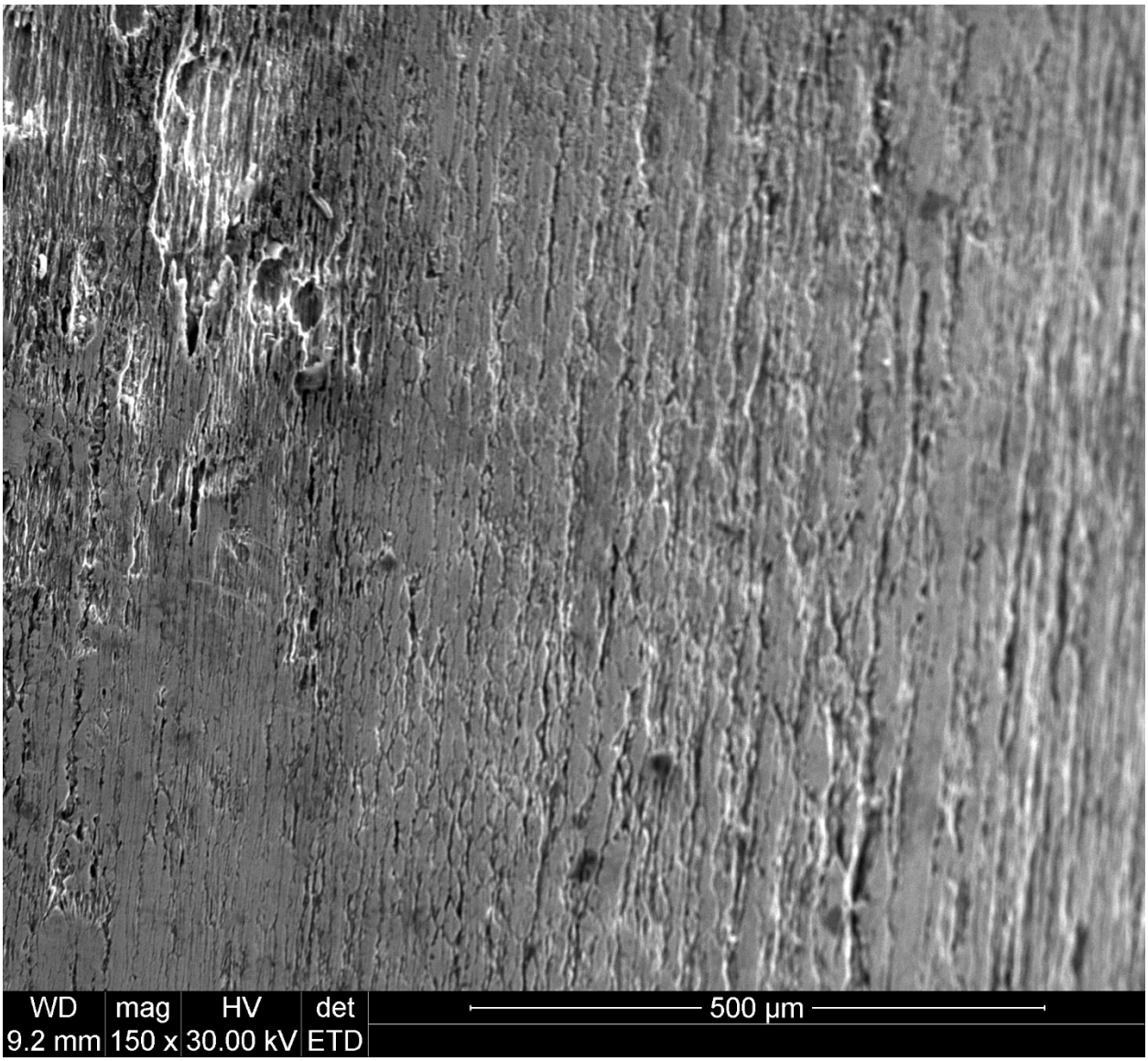


Figure 130. Scanning electron microscope images for site 3 (Nontribos fungi mix)

Figure 130 shows a different location at 150X where there is a corrosion site on the left, but only minor damage on the rest of the area. Figure 131 and Figure 132 shows images taken at high magnification presenting different structures when compared to those described above. Figure 132 shows more clearly the needle-like crystalline structure at the bottom, the magnification in this case is 10,000X. It is not known if the needles are corrosion products not removed while cleaning or if these grew after cleaning during the time that elapsed before cutting off the small two to three centimeter section (which was about two to three weeks). The wires were kept in ziplock bags in a low humidity chamber.

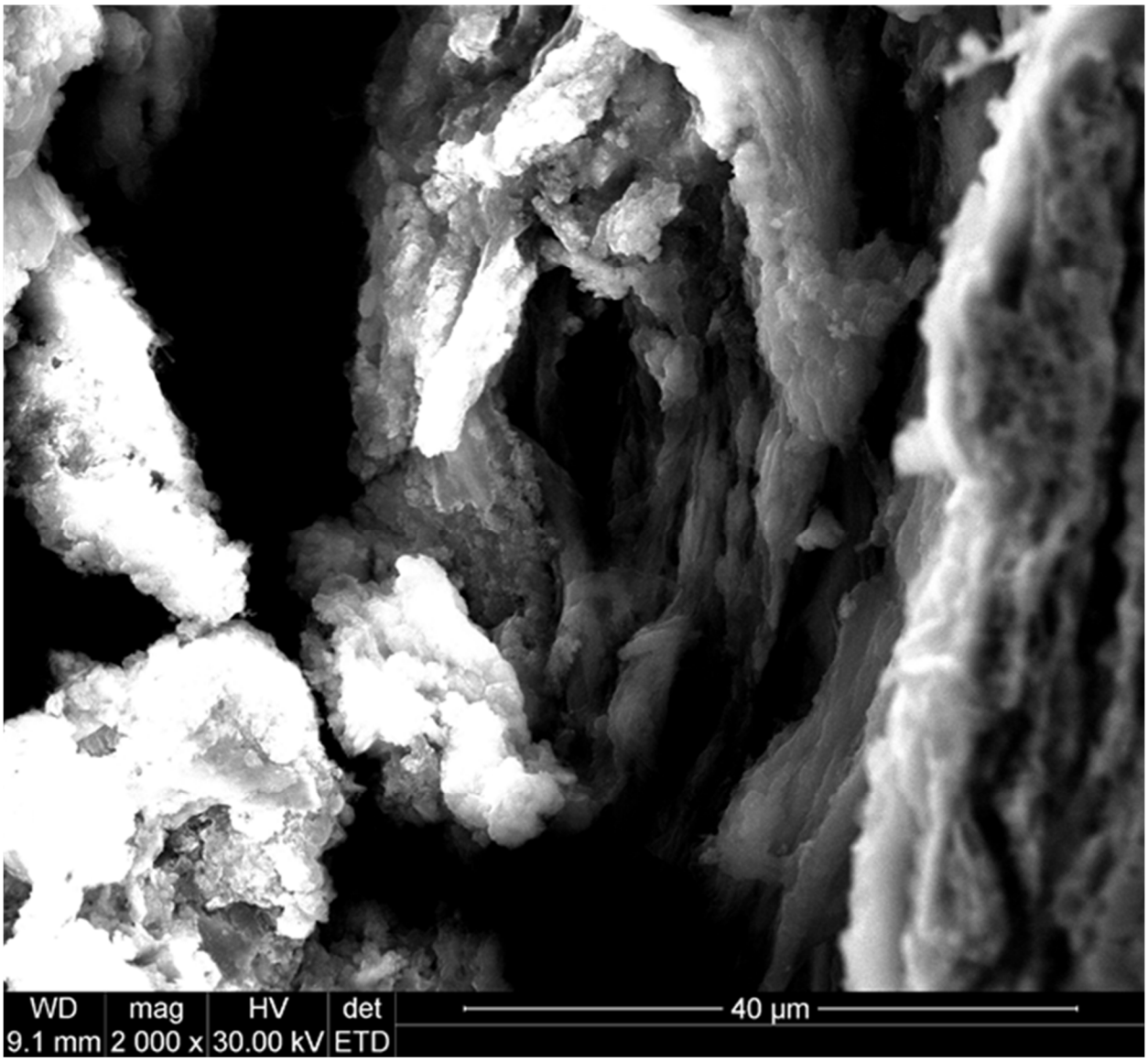


Figure 131 Scanning electron microscope images for site 4 (Nontribos fungi mix)

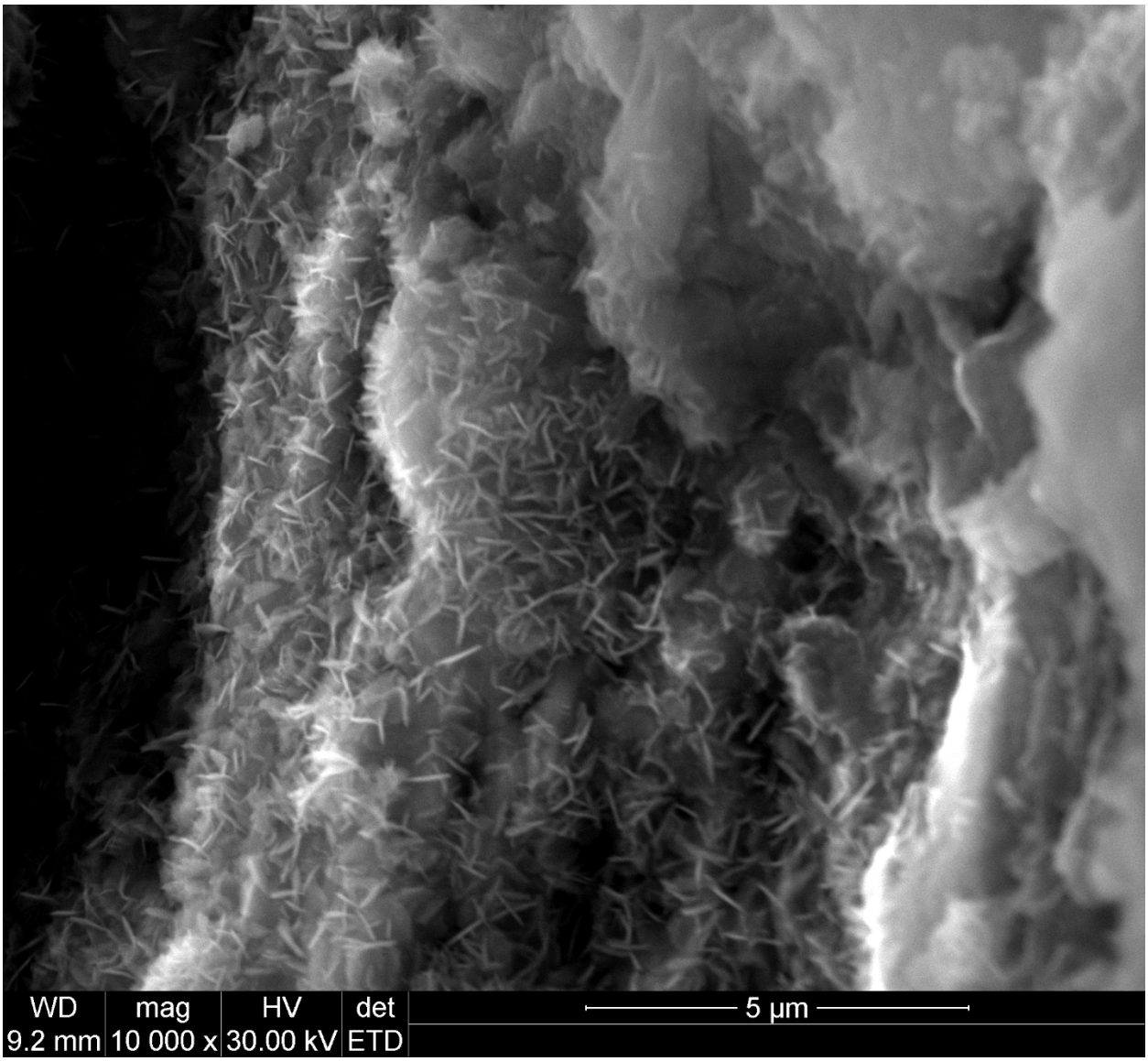


Figure 132 Scanning electron microscope images for site 5 (Nontribos fungi mix)

5.4 Nontribos-coated Strand Sprayed with Water

A strand was coated with Nontribos and sprayed with water, it was stored in a PVC pipe and placed outdoors. Figure 133 shows cleaned wires (top three still with mineral spirits) for the section of the strand cut off. No significant amount of corrosion was observed from this view. A section was cut off from one of these wires that appeared to have some corrosion. Images were taken with the SEM and are shown in Figure 134. The low magnification image (26X) shows that there are large areas with no corrosion and a few small spots with corrosion. The 300X image shows that corrosion took place and it displays striation like patterns. The higher magnification images show additional detail.

Figure 133. . Section of Nontribos coated strand, after cleaning – (water)

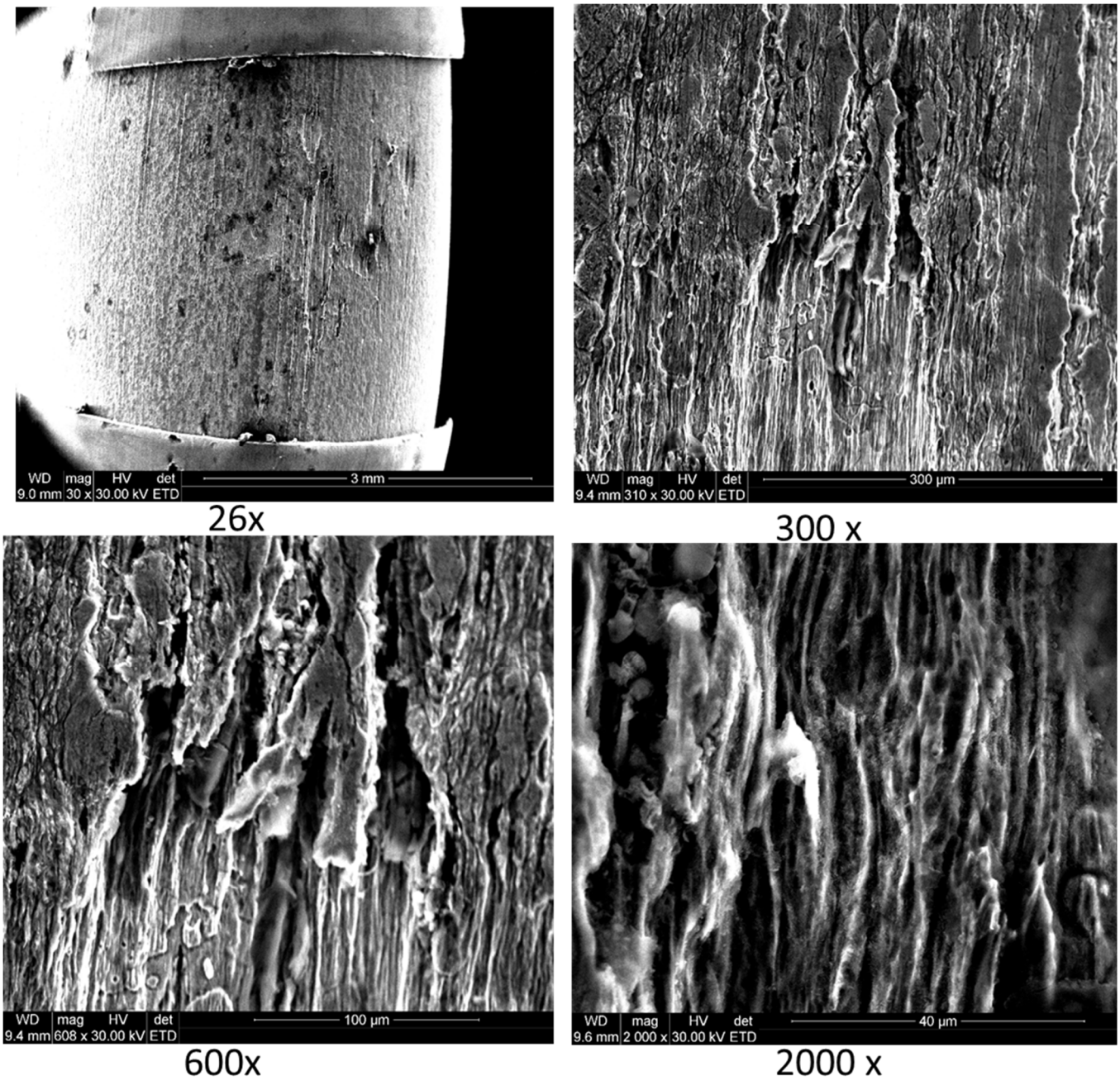


Figure 134. Scanning electron microscope images for site 1(Nontribos water)

5.5 Civetea-coated Strand Sprayed with Water

Three wires from the cut off section were cleaned for the strand coated with Civetea and later sprayed with water. No SEM images were taken. Figure 135 shows the seven wires, three of which were chemically cleaned. Figure 136 shows a picture of a section with a corroding site on the top row. The bottom row shows three pictures taken with the stereo microscope. The 3X images indicate that pitting like corrosion took place in some areas.

Figure 135. Section of Civetea coated strand, after cleaning (water)

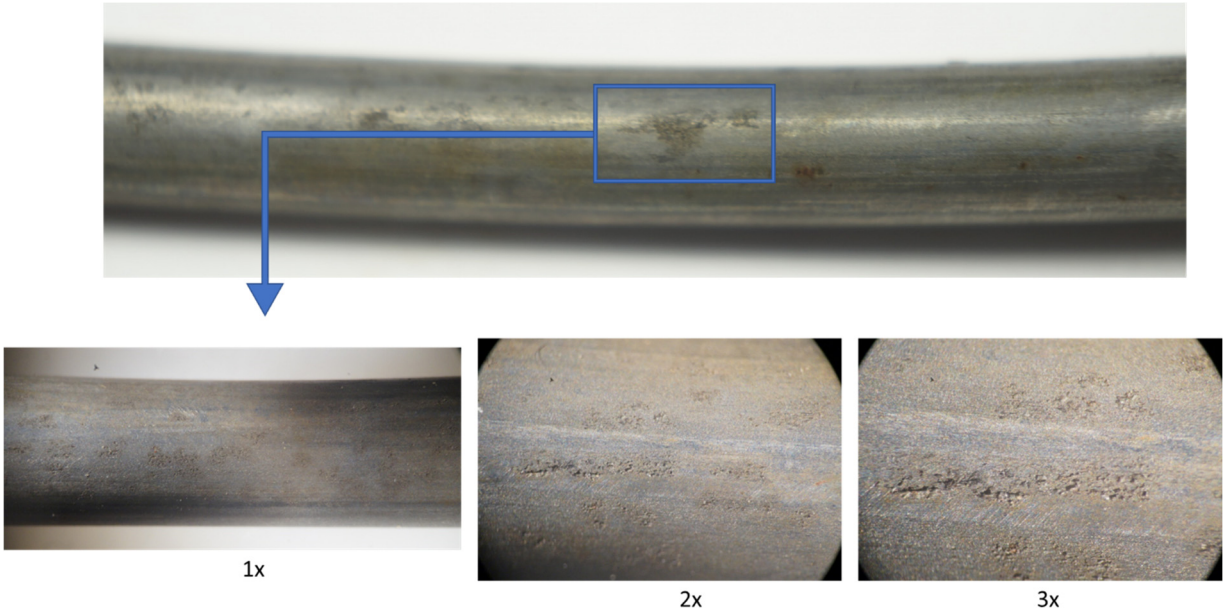


Figure 136. Close-up selected wire, after cleaning (Civetea water)

The corrosion sites appear to be more pronounced on the strands that were sprayed with fungi, regardless of the flexible filler present. The outdoor exposure likely allowed the sprayed solution

to evaporate within a short period of time. This might explain why the location of the corroding sites were sporadic.

5.7 Small Injected Samples –Inoculated with Fungi

Polycarbonate tubing with 0.75 inch ID (inner diameter) and 1 inch OD (outside diameter) and six to eight inches in length were injected with each of the available flexible fillers. Two or three wires were inserted on each tube prior to injecting the filler. Black caps fitting the tube were placed on both ends. Figure 137 shows a picture for a couple of samples. Figure 138 shows a top view section for three of the samples with the cap removed. As indicated in the experimental section different solutions (water/control, water with fungi mix, *Aspergillus flavus* (AF), *Fusarium oxysporum* (FO), *Penicillium chrysogenum* (PC)) were sprayed from one end of the samples.

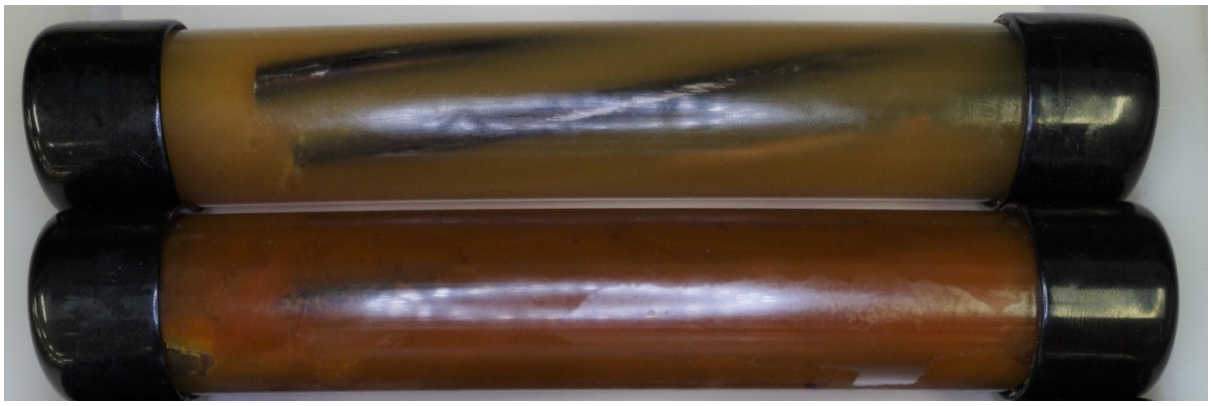


Figure 137. Pictures showing two of the 6-inch long samples.

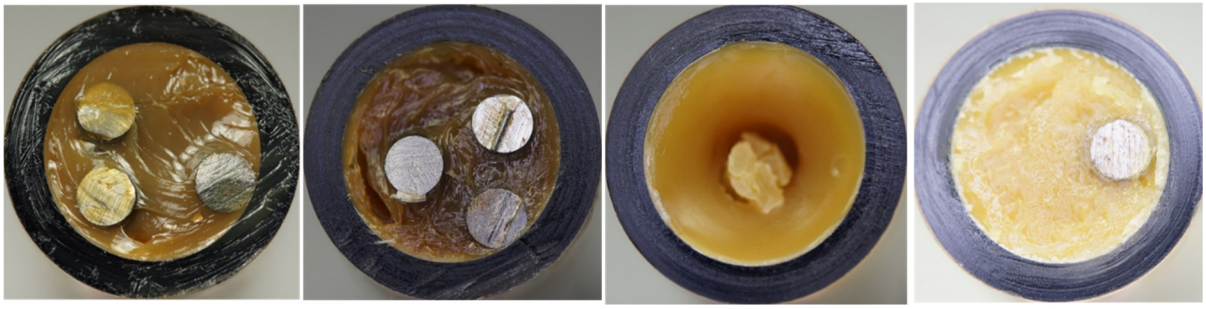


Figure 138. Pictures showing the cross-section after removing the cap.

After several months of exposure and periodic spraying (the first time 0.2 milliliter with fungi mix, and later sprays were with tap water except for the samples sprayed with fungi mix which continue to be sprayed with fungi mix). With time, some of the wires started showing rust spots. These were identified as part of the periodic visual inspections. The pictures below show two examples. Figure 139 shows a couple of examples for single wires that were close to the surface and showed corrosion spots at the surface visible thru the polycarbonate. Upon removing the caps, rust was visible from one end, but not from the opposite side (not shown here).

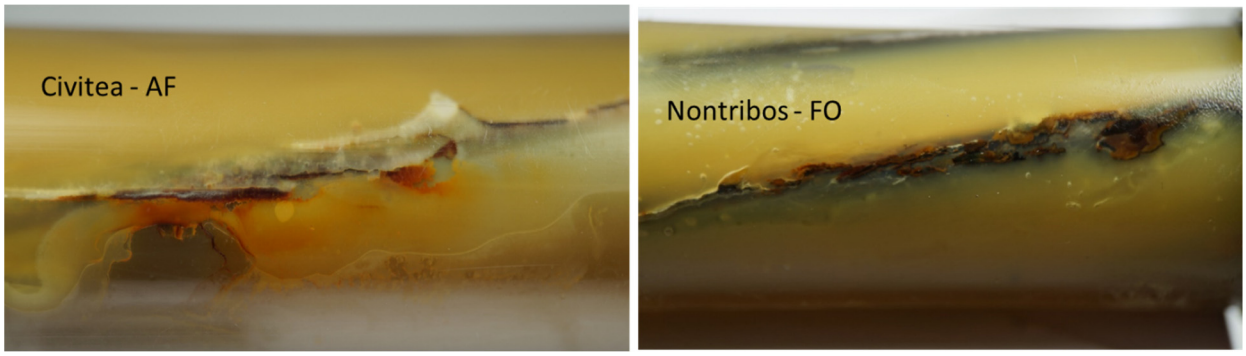


Figure 139. Example of wires that showed rust spots after several months of exposure.

The following pages will show the samples grouped per solution sprayed. Pictures were taken from two different views, showing that not all samples showed rust spots. The wires were not always close to the surface. Figure 140 shows the samples sprayed with solutions containing *Aspergillus flavus* (AF). Figure 141 shows the samples sprayed with solutions containing *Fusarium oxysporum* (FO). Figure 142, Figure 143, and Figure 144 show the samples sprayed with solutions containing *Penicillium chrysogenum* (PC), fungi mix, and water respectively.

Figure 140. Two views of the five samples sprayed with *Aspergillus flavus*.

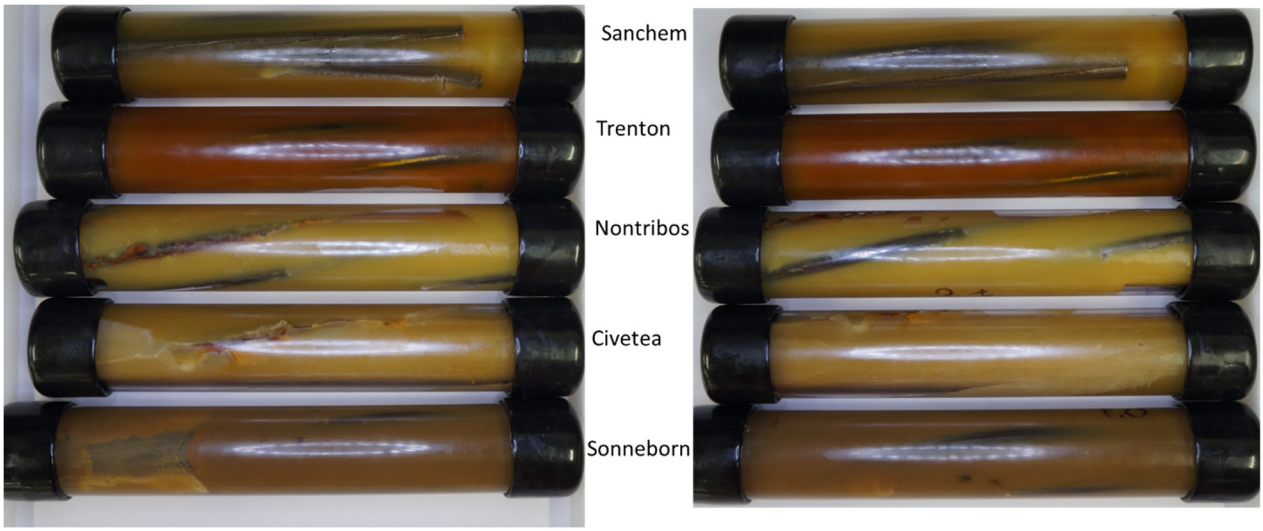


Figure 141. Two views of the five samples sprayed with *Fusarium oxysporum*.

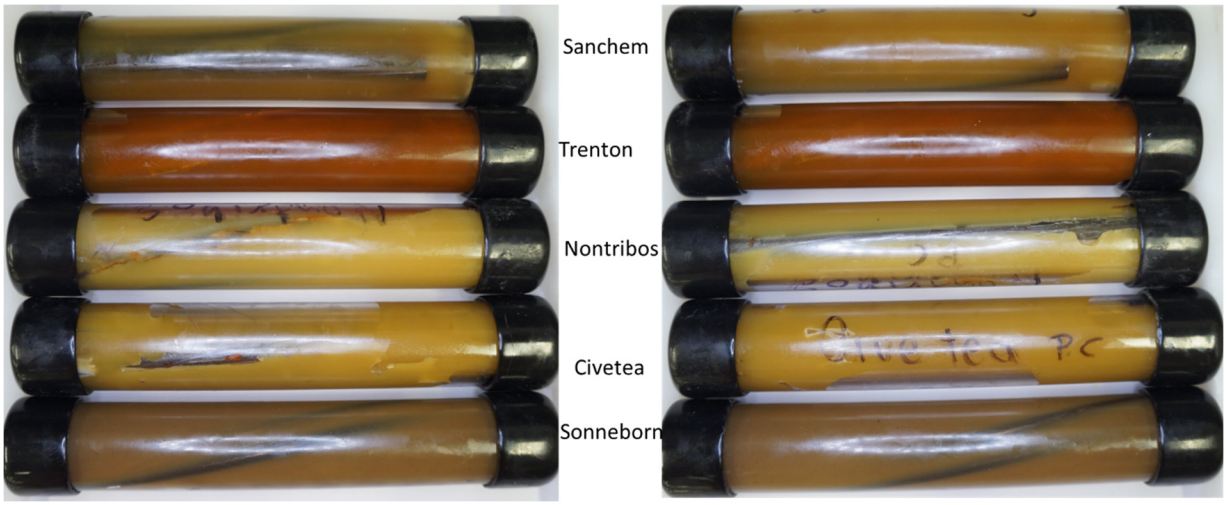


Figure 142. Two views of the five samples sprayed with *Penicillium chrysogenum*

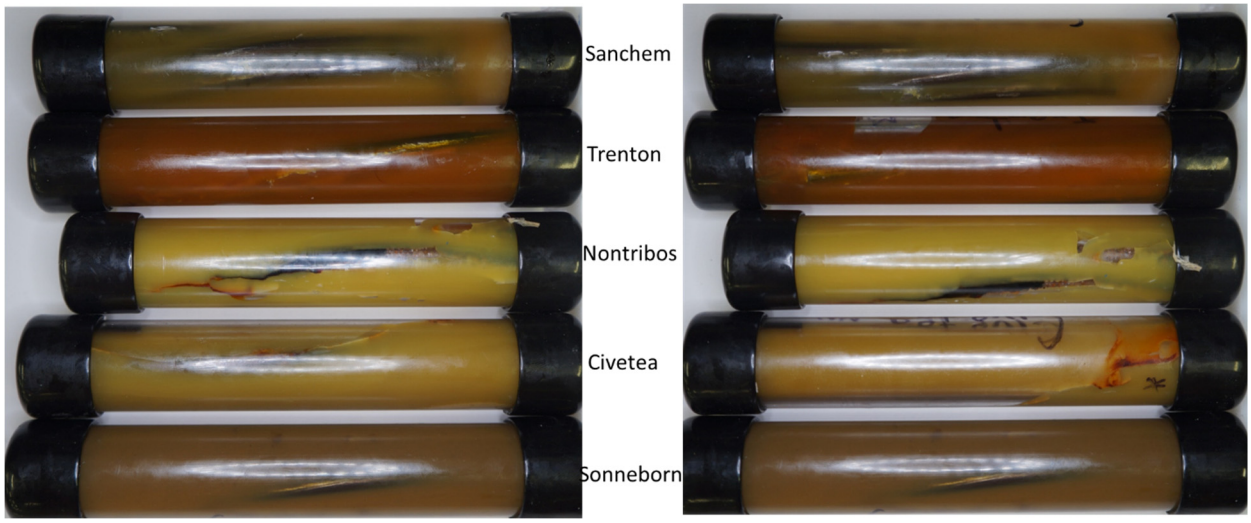


Figure 143. Two views of the five samples sprayed with fungi mix.

Figure 144. Two views of the five samples sprayed with water.

Figure 140 to Figure 144 show the corrosion spots visible on wires close to the surface on samples injected with Civetea and Nontribos and sprayed with a specific fungi or with fungi mix, but no corrosion spots were visible on those sprayed only with water. Recall that samples sprayed with *Aspergillus flavus*, *Fusarium oxysporum*, *Penicillium chrysogenum* were done only on the initial spray and later sprays were done using water. For the samples sprayed with fungi mix, the same solution was used during later sprays. Corrosion spots were not visible through the polycarbonate tube on the samples injected with Sanchem, Trenton or Sonneborn.

A wire from each of the samples shown above was removed 200 days after initial spray. The wires were then cleaned and photographs were taken. The section below shows the wires after cleaning grouped per filler type. Then, selected close-ups with the camera and the stereo microscope are presented. For selected cases, a smaller piece was cut off and then placed in the SEM for higher magnification analysis. Figures showing some of the images taken are also included.

5.7.1 Civetea

Figure 145 shows the wires removed from samples injected with Civetea. Very modest amounts of corrosion were observed upon removing the flexible filler and then cleaning the wires. Figure 146 shows close-ups images after cleaning of the wire covered with Civetea and sprayed with water. The SEM photographs show that a modest amount of corrosion took place. The SEM pictures were taken a few weeks after cleaning. The 10,000X image shows a number of small crystals, that are likely due to corrosion products that developed after cleaning. Figure 147 shows close-ups images of the wire covered with Civetea and sprayed with FO and later sprayed with water (after cleaning). The low magnification image shows a large corrosion spot, the intermediate magnification (150X and 300X) shows striations due to corrosion. More detail of the striations are visible on the images taken at 600X and 1,000X. Figure 148 shows close-up images of the wire covered with Civetea and sprayed with fungi mix. The corrosion extent observed on the wire sprayed with fungi mix is intermediate of that observed on the other two wires just described. The higher magnification images show that striations are present but not as many. The 10,000X image shows needle like particles at the bottom of the striations.

Figure 145. Wires after cleaning removed from samples injected with Civetea.

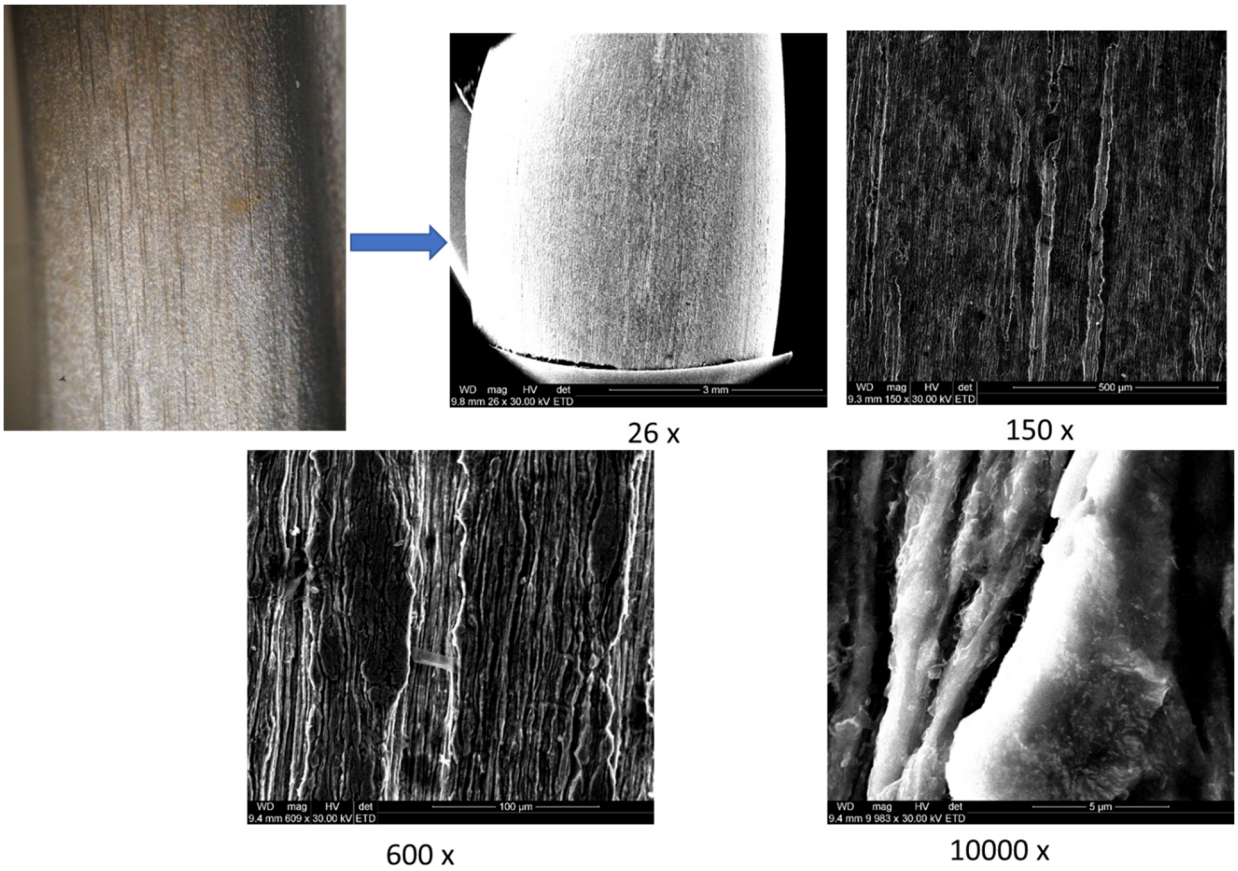


Figure 146. Stereo microscope and scanning electron microscope images. (Civetea – water).

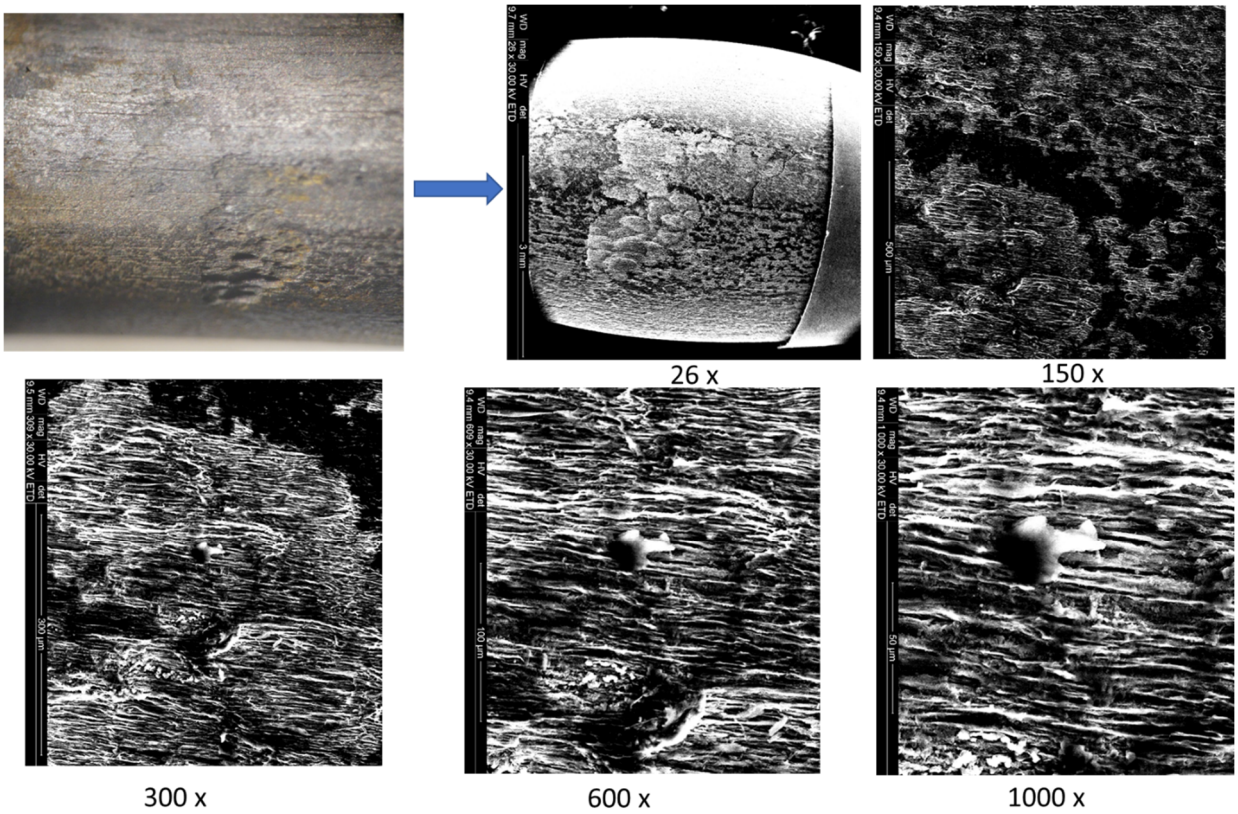


Figure 147. Stereo microscope and scanning electron microscope images (Civetea sprayed w/FO).

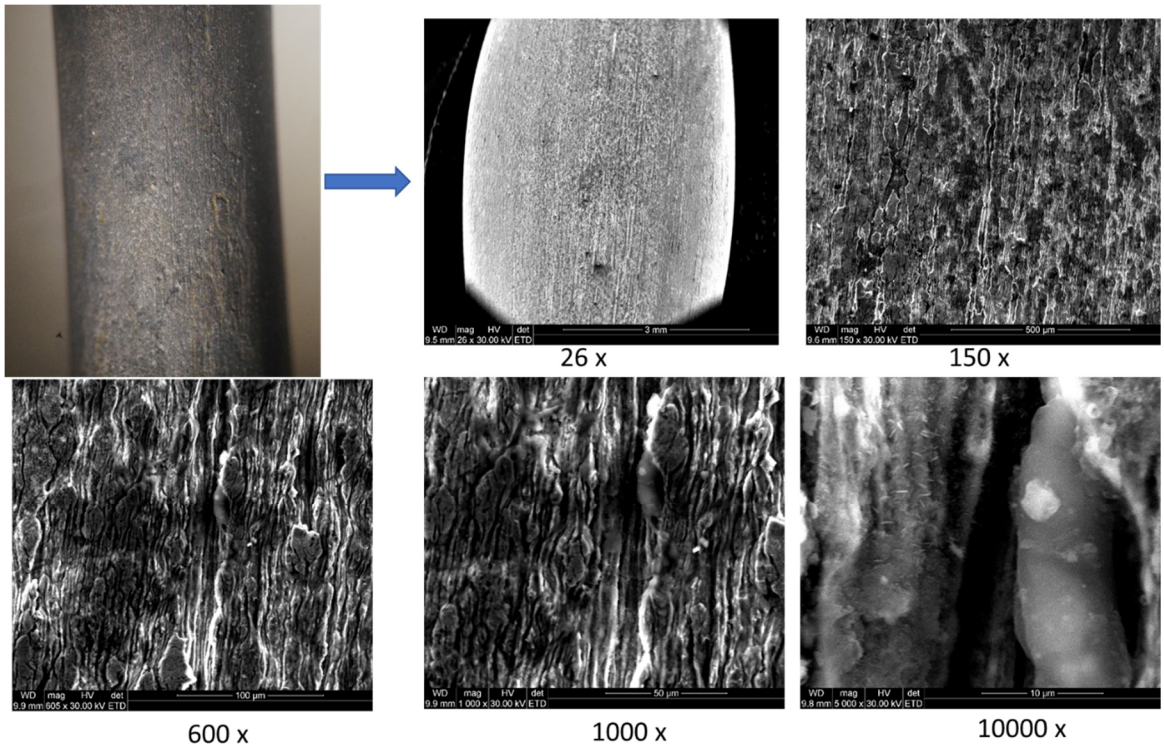


Figure 148. – SEM images of wire after cleaning (Civetea sprayed w/fungi mix)

5.7.2 Nontribos

Figure 149 shows the wires removed from samples injected with Nontribos. Very modest amounts of corrosion were observed upon removing the flexible filler and then cleaning the wires. Figure 150 shows close-up images after cleaning the wire covered with Nontribos and sprayed with water. The top left image shows a stereo microscope picture. The other images on this figure were taken with the SEM at various magnifications ranging from 26X to 10,000X. The SEM photographs show that a modest amount of corrosion took place. A crystalline structure appears to have developed at the surface of the sample (10,000X magnification). Figure 151 shows close-up images after cleaning of the wire covered with Nontribos and sprayed initially with FO and later sprayed with water. The low magnification image shows a shallow corrosion spot, the intermediate magnification (150X and 300X) shows that striations due to corrosion are visible, and more detail of the striations are visible on the images taken at 600X and 1,000X. Figure 152 and Figure 153 show close-up images of the wire covered with Nontribos and sprayed with fungi mix for two different sites. Figure 152 shows site 1 and there are several spots where corrosion took place. The higher magnification images show striations within the corroding spots. Figure 153 shows site 2 and that there were two depths on the corrosion products. Both depths are observable up to 300X. At higher magnifications the striations are observed and they are somewhat different than that observed on the wires injected with Civetea and sprayed with fungi mix.

Figure 149. Wires after cleaning removed from samples injected with Nontribos.

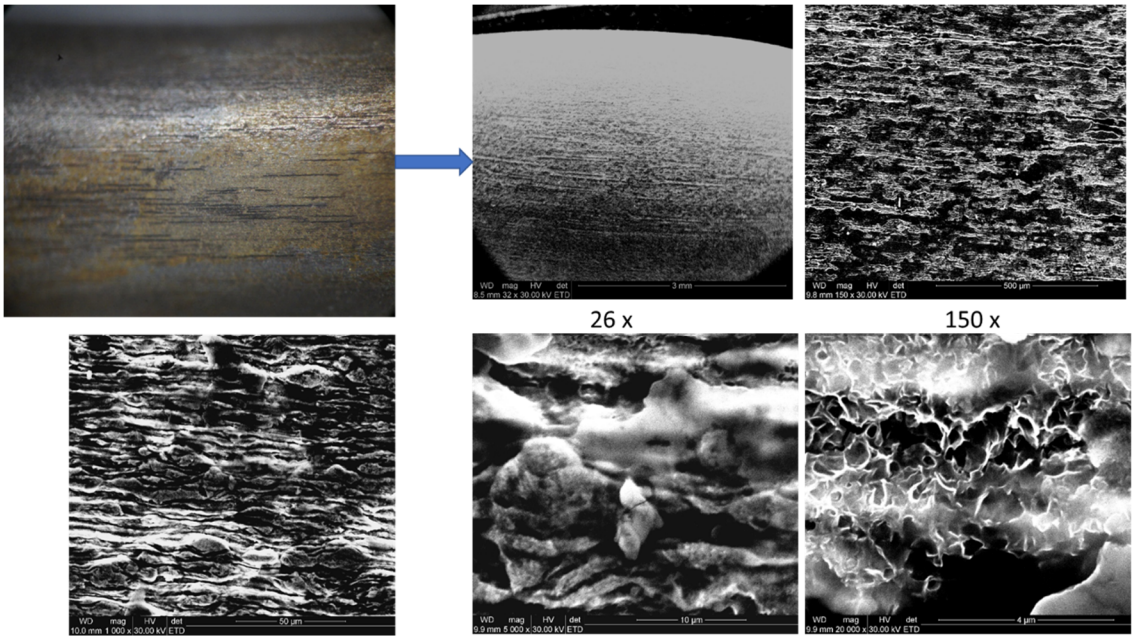


Figure 150. SEM images of wire after cleaning (Nontribos – water)

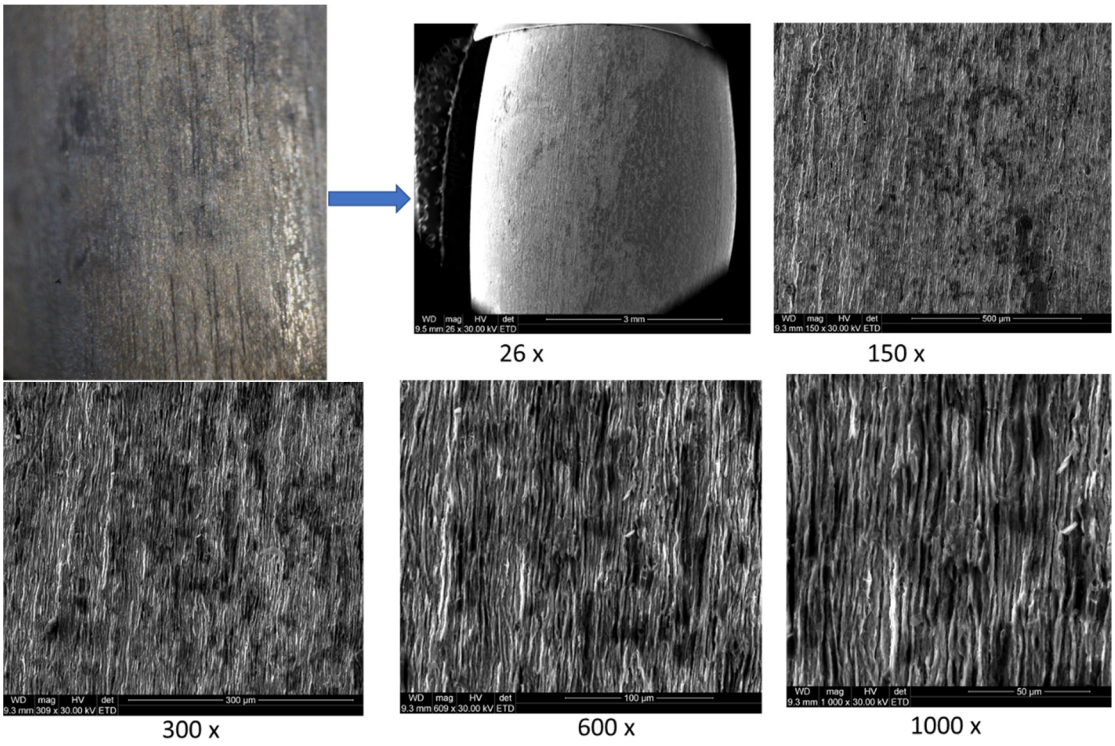


Figure 151. SEM images of wire after cleaning (Nontribos – FO).

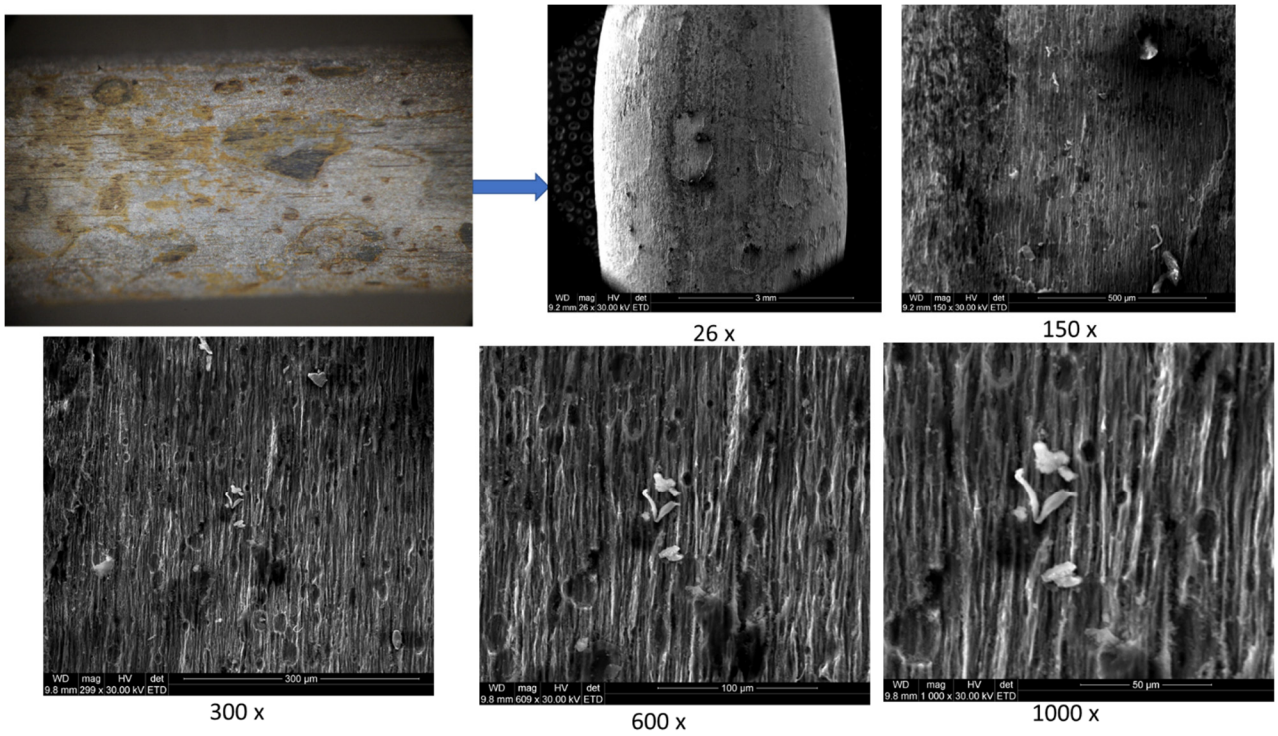


Figure 152. Site 1: SEM images of wire after cleaning (Nontribos fungi mix)

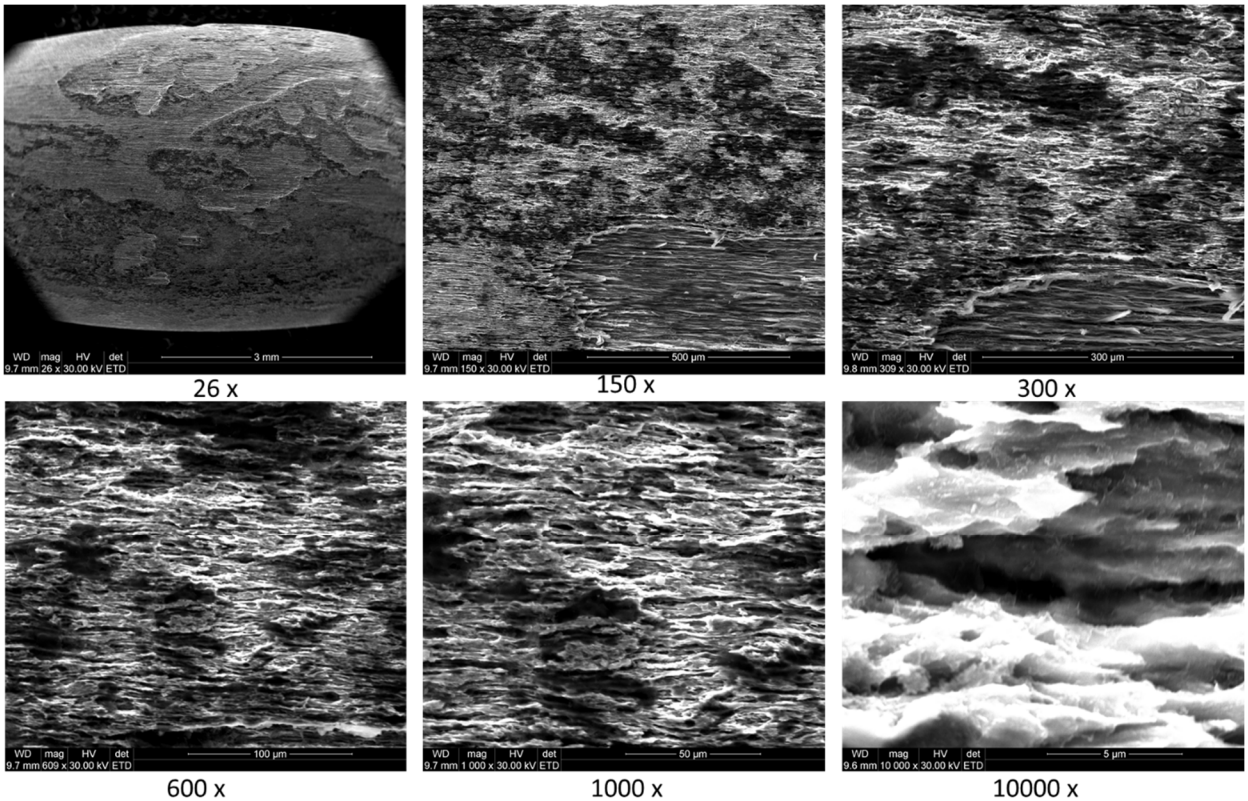


Figure 153. Site 2: SEM images of wire after cleaning (Nontribos fungi mix)

The wires cleaned for samples injected with Sanchem, Sonneborn, and Trenton were those sprayed with fungi mix.

5.7.3 Sanchem

Figure 154 shows an image after cleaning of the wire coated with Sanchem, Figure 155 show images taken with the SEM. The images of the selected location show a different pattern than previously. The low magnification suggests no corrosion. The higher magnification images suggest that corrosion might have taken place, but no significant penetration occurred.

Figure 154. Wire after cleaning removed from samples injected with Sanchem – sprayed with fungi mix

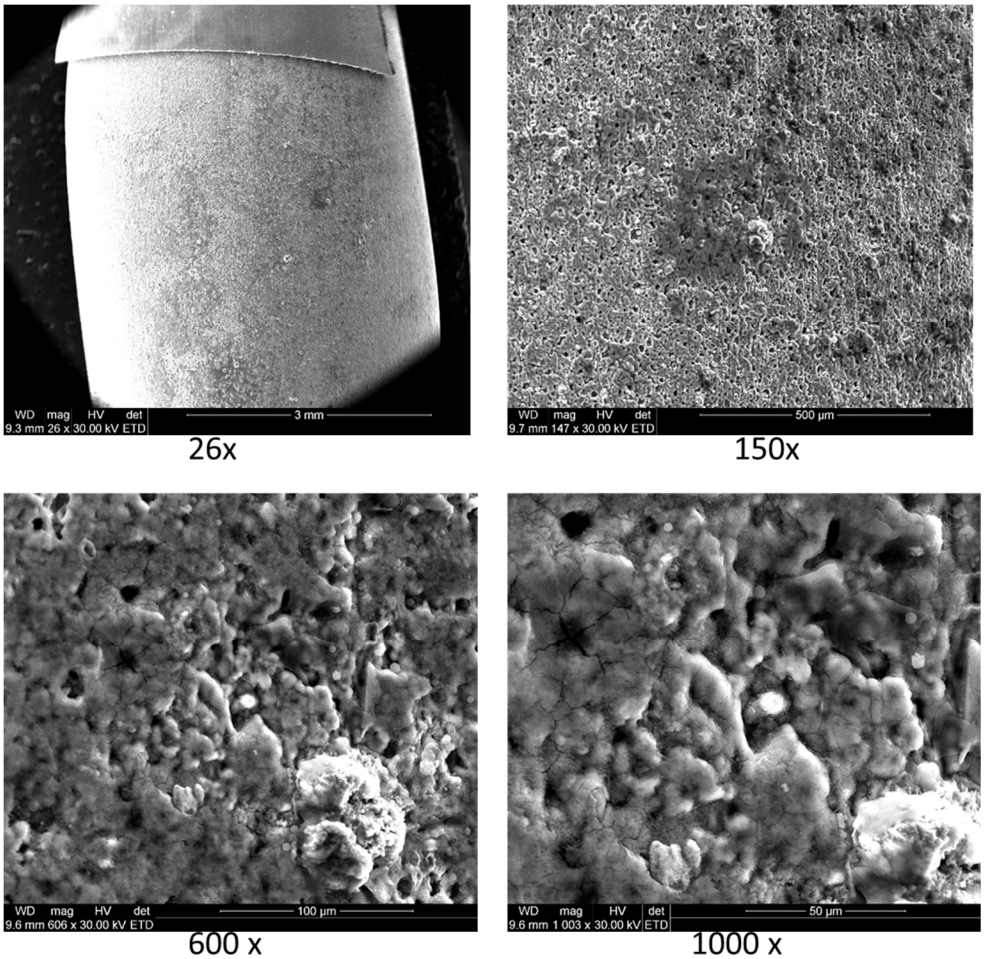


Figure 155. Site 2: SEM images of a wire after cleaning exposed covered w/Sanchem – sprayed with fungi mix.

5.7.4 Sonneborn

Figure 156 shows a wire removed from a sample injected with Sonneborn and sprayed with fungi mix. No corrosion sites were observed. Figure 157 shows picture taken with a stereo microscope and five pictures taken with the SEM. The low magnification (26X and 150X) suggest that there is some damage at the surface. Higher magnification shows that part of the surface did not corrode and that at certain regions, there were stripes with striations, but these appear to be shallow.

Figure 156. Wire after cleaning removed from samples injected with Sonneborn – sprayed with fungi mix.

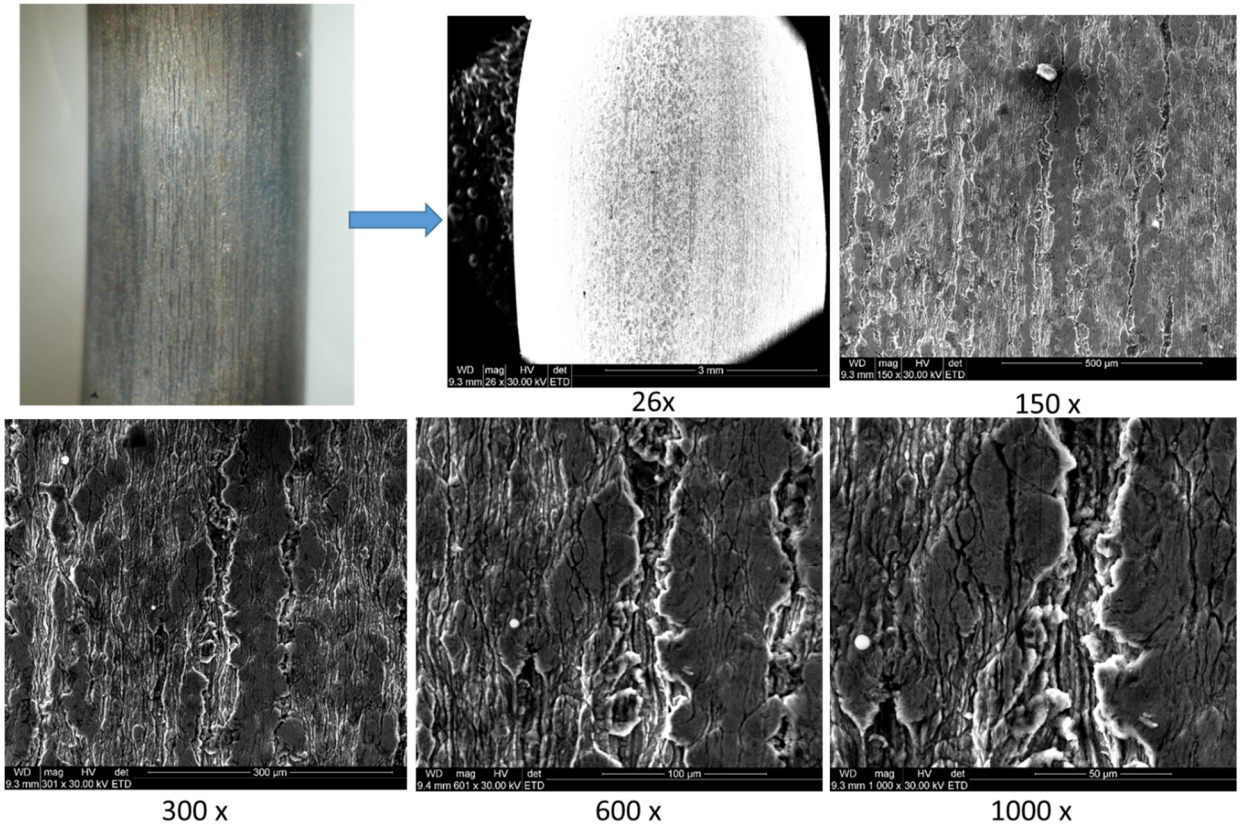
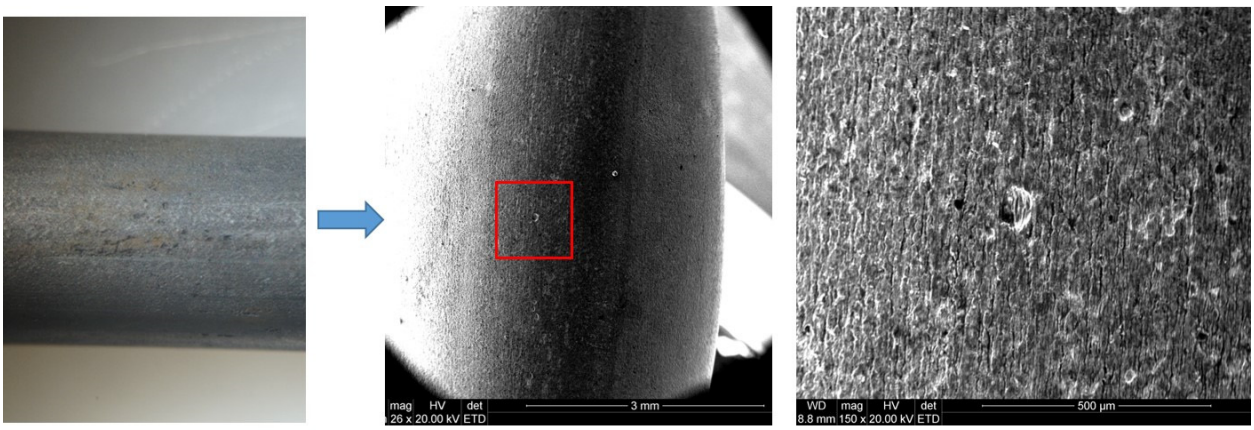


Figure 157. Close-up of wire after cleaning removed from samples injected with Sonneborn sprayed with fungi mix

5.7.5 Trenton

Figure 158 shows a section of the wire removed from the sample injected with Trento and sprayed with fungi mix. Very few corrosion sites are observed. Figure 159 shows a picture taken with a stereo microscope and four pictures taken with the SEM. The low magnification (26X and 150X) shows a small pit on the assessed area. The higher magnification images show striations. Similar small corroding sites were observed at other locations within the piece placed on the scanning electron microscope.

Figure 158. Wire section after cleaning for wire removed from samples injected with Trenton – sprayed with fungi mix.



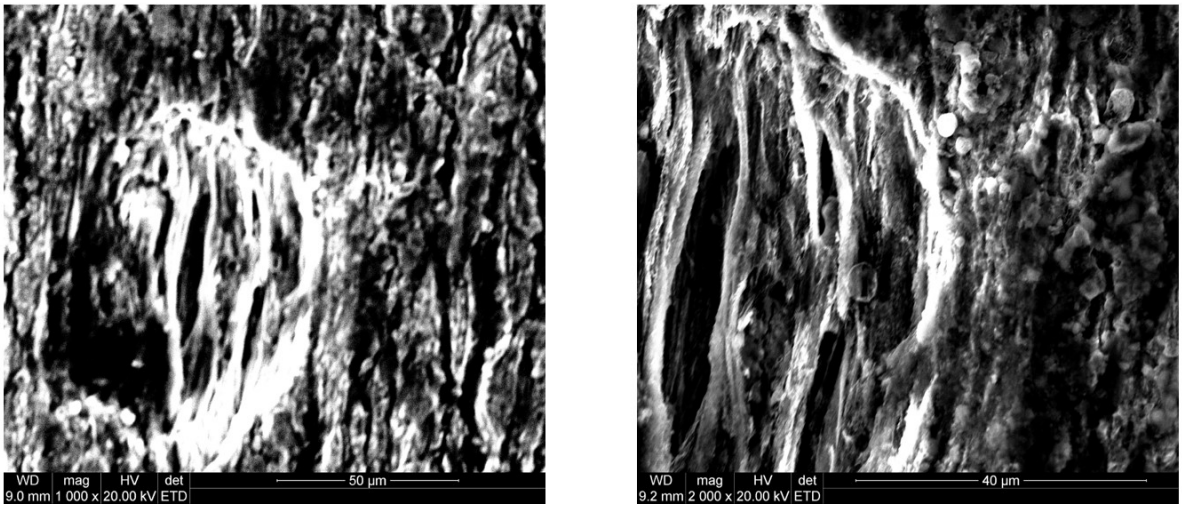


Figure 159. Close-up of wire after cleaning removed from samples injected with Trenton sprayed with fungi mix

Chapter 6 - Discussion

6.1 Fillers

6.1.1 Color Change

The Civetea filler exposed outdoors at the semi-sheltered location experienced color change from a yellow to a dark brown. This was also observed after several months on the samples where Civetea was injected into polycarbonate tubes and exposed at the semi-sheltered location.

6.1.2 Inoculated Fillers

The different fillers were sprayed with each of the fungi and the fungi mix. The inoculation was applied only once and the samples were stored in the lab humidity and temperature. The fungi grew in some of the fillers but not on all. For example after 25 days visual evidence was found on the Trenton filler sample sprayed with *Aspergillus flavus*. *Fusarium oxysporum* growth was observed after 34 days on Sonneborn filler. However, after inspecting the inoculated filler samples after two years the area covered with fungi was smaller, likely because no additional moisture was introduced.

6.2 Coated Strands and Coated Wires

6.2.1 Coated Strands and Coated Single Wires Exposed Outdoors

General corrosion was observed on the single wires after cleaning, for both coated strands and single coated wires exposed outdoors at the semi-shelter location. The direct exposure to particulates from ocean spray, wetting when rain and wind reached the samples, and the temperature and humidity of the site contributed to breaching of the coating. The corrosion spots did not cover the whole surface for the cleaned, coated steel strands. Similarly, the corrosion spots did not cover the whole area of the single coated wires exposed outdoors. The most corrosion was observed on the wires coated with Nontribos, followed by the strand covered with Civetea.

Minor corrosion was observed after cleaning wires exposed at the partially closed container placed outdoors.

6.2.2 Coated Single Wires Exposed Indoors

The coated wires exposed in the elevated temperature room and high humidity showed modest amounts of corrosion. For wires exposed to room temperature and high humidity the worst corrosion was observed on the blank samples (wire that was not coated), followed by wire coated with Nontribos. Similar amounts of corrosion were observed after cleaning the wires coated with Civetea and Sonneborn. Minor corrosion was observed on the wires coated with Trenton and Sanchem. There were regions with no corrosion on all the wires. Trenton and Sonneborn coated wires appear to have the least amount of corrosion for the coated wires that were exposed to high humidity at room and elevated temperature, respectively.

6.2.3 Coated Wires Contaminated with Fungi

Samples sprayed with 1.5 milliliter of solution.

The coated wire specimens that were sprayed with fungi mix presented the most corrosion in most instances. With respect to the samples sprayed with one type of Fungi, it was observed that the

specimens sprayed with *Fusarium oxysporum* presented the most corrosion, followed by the samples sprayed with *Aspergillus flavus* and finally the wires sprayed with *Penicillium chrysogenum*. These results are consistent with the results obtained on fillers sprayed with the different fungi, where the fillers sprayed with *Fusarium oxysporum* and *Aspergillus flavus* were able to use the flexible fillers as a carbon source and developed on them.

Samples sprayed with 0.6 milliliter
Significantly less corrosion extent was observed after cleaning on the wires sprayed with 0.6 milliliter compared to the wires sprayed with 1.5 milliliter. The better performance could in part due to the method used to apply the filler onto these wire.

0.75 inch diameter – polycarbonate specimens
The five fillers investigated were injected into 0.75 inch diameter polycarbonate and then sprayed with water, each fungi (and later with water) and with fungi mix. Some of the embedded wires ended up next to the polycarbonate surface, which allowed the sprayed solution to wick along the wire surface. Most of the wires at these locations developed corrosion. It is possible that the injected filler was not able to coat the whole wire surface.

After cleaning selected samples were observed with in the SEM for large magnification. Cross-section loss was observed on several wires. The wires coated with Civetea appear to experience the most corrosion.

Single coated strand sprayed with fungi mix or water (exposed outdoors)
Strands coated with Civetea, Nontribos and Trenton were subjected to the outdoor environment. After 280 days, a section was cut off, then the filler was removed. Selected wires were cleaned using ASTM G01. The wires that showed the worst corrosion had smaller (< 2 centimeter long) sections cut off and observed under the scanning electron microscope. The sample sprayed with fungi mix coated with Nontribos showed the most corrosion, followed by the wires coated with Civetea. However, even the strand coated with Trenton sprayed with fungi mix showed a few corrosion spots. Most of the corrosion sites were not significantly deep (a few tend to hundred microns at most). A large surface of the wire showed no corrosion. Thus, the corrosion was localized. There were likely regions with a thin layer of the filler that the sprayed fungi mix solution was able to penetrate over time. In general, the worst corrosion was observed on the samples sprayed with fungi mix (when compared to the strands sprayed with water).

6.3 Filler Injected Samples (Larger Samples)

6.3.1 Four-foot-long Specimens – HDPE (4-inch diameter)

The available samples were injected with Trenton at SRC. These samples were exposed outdoors at the west side of the property under the stairs. The samples were periodically sprayed with water, each fungi (the first time and later with water), and two samples were sprayed with fungi mix. The cuts done to bring the samples to size in some cases caused darkening of the strands. In general, minor corrosion was observed on a few wires, but most of the strand ends (i.e., the cut off surface) showed no corrosion. On most specimens, about half of the strands had a layer of the filler covering the steel wires ends, which likely allowed for strand protection. A few wires showed corrosion

during the May 2018 inspection. The filler appears to have partially re-melted when compared to the pictures taken in December 2017.

6.3.2 Four-foot-long Specimens – HDPE (2-inch diameter)

No major corrosion progression was observed on these specimens. Minor corrosion was observed on the wire ends. Most wires had a layer of the filler covering them. The filler did move some over time and as a result of warmer seasonal temperatures.

6.3.3 Four-foot-long Specimens – Polycarbonate (Outdoors Semi-sheltered Location)

Modest corrosion was observed on a few wire ends. These wires likely had a thin layer of the filler covering them (or just partially covering them). Water wicking was observed in a few occasions. The lower end of the sample showed moisture, but the solution was sprayed on the opposite end of the sample (higher side). Both ends were removed periodically to document changes. Nontribos and Civetea samples sprayed with fungi mix developed corrosion on a few of the wires.

6.3.4 Two-foot-long Specimens – Polycarbonate (Exposed to Laboratory Conditions)

Corrosion was observed on a few of the wire-ends (cut off) that either did not have a filler layer or had a very thin layer to start with. The corrosion spots grew as time passed. Indoors, the temperature and humidity in the room is controlled most of the time (except on occasions when the air-conditioner stopped working). It likely that the water did not evaporate as fast as for the samples exposed outdoors. The corrosion spots appear to be superficial, however no segment was cut off from these samples.

Chapter 7 - Conclusions

7.1 Small Samples

In this study, the corrosion performance of PT steel strands and single wires coated with five different flexible fillers was investigated. The performance of flexible fillers after contamination with three different fungi species was also examined. Exposure tests, contamination tests, and electrochemical corrosion tests were designed and carried out. To further examine the surface conditions of the steel strand, microscopic observation was performed. After analysis and discussion of experimental results, the following conclusions can be drawn:

(1) For exposure test, single wires show worse corrosion than the steel strand due to complete exposure of the whole surface to the testing environment. For most of the coated strands, corrosion started at the ends due to thinner or no filler present at this locations edge. The corrosion performance of the fillers depends on the filler layer thickness and uniformness after application. For single wire exposure tests, rust started to be observed after 20 days, 43 days, 49 days, 114 days and 126 days of exposure for wires coated with type 1, type 2, type 3, type 4, and type 5 filler, respectively. Particulates and moisture likely penetrated the coating and allowed corrosion to initiate.
(2) For the fungi contamination test, a single wire coated with a filler sprayed with the fungi mix (a suspension with the three fungi) showed the worst corrosion. Among the five fillers, the wires coated with Sonneborn (type 5) filler show less corrosion attack than the wires coated with the other fillers.
(3) With regard to the electrochemical corrosion test, steel strands coated with type 3 filler (Civetea) showed corrosion after approximately 14 days of immersion in soil solution with different pH values. Steel strands coated with type 4 and type 5 also show signs of corrosion after about five months of immersion testing. Steel strand coated with type 1 and type 2 did not show any signs of corrosion from the beginning of the test. These tests show the corrosion protection from water and ion penetration because the soil solution did not contain fungi nor bacteria.

For samples injected (0.75 inch diameter polycarbonate tube) with the different fillers and then sprayed with: Water, (FO/AF/PC) one time and then water, fungi mix

- Corrosion was observed on wires sprayed with fungi mix. The worst damage was observed on wires for samples injected with Nontribos and Civetea. Wires coated with other fillers showed less corrosion (smaller sites and minor damage were observed)
- Wires from Civetea and Nontribos injected samples sprayed with water or sprayed with (FO/FA/PC) one time and then water also showed corrosion, but to a lesser extent compared to the samples sprayed with fungi mix.

7.2 Large Samples

For samples injected with the different fillers and then sprayed with: fungi mix, water, or (FO/AF/PC) one time and then water:

- Little or no corrosion was observed on four inch diameter samples
- Little or no corrosion was observed on two inch diameter samples

For single coated strand samples sprayed with fungi mix (stored in PVC outdoors) or water

- The worst damage was observed on the samples coated with Nontribos filler. Civetea and Trenton coated strands showed less corrosion.
- Corrosion initiated on the strand coated with Nontribos and sprayed with water, but moderate corrosion damage was observed when compared to the strand coated with Nontribos and sprayed with fungi mix.

7.3 Recommendations

- Ensure that a proper seal takes place at anchorages and other locations where moisture (and/or outside particles) could enter to where the filler-coated strands are. Water accumulation needs to be prevented (previous reported failures were in part caused by this).
- Proper care when handling to ensure coverage of both strands and flexible fillers prior to and during injection to prevent contamination and to prevent moisture to enter the ducts.
- Inspections for moisture marks (on the concrete) could be used as a monitoring tool during the life of the bridge. Additional a moisture sensing device, could assist in detecting if water uptake has occurred at anchorages or the lower points of the PT systems that are injected with flexible filler.

References

[1] M. Catalá and D. Quigley, *FDOT Structures Manual (SDG 1.11.5)*, vol. 1, no. 625. 2016.

[2] FDOT, *FDOT's Standard Specifications for Road and Bridge Construction (Section 938)*. 2017.

[3] W. Podolny, W. R. Cox, J. M. Hooks, M. D. Miller, A. J. Moreton, M. A. Shahawy, D. Edwards, M. Madani, R. K. Montgomery, B. Pielstick, and M.-C. Tang, "Performance of Concrete Segmental and Cable-Stayed Bridges in Europe," 2001.

[4] M. W. Holley, "Continuous acoustic monitoring of prestressed structures. In: Corrosion and its control in the new millennium," in *Proceedings of the NACE International Northern Area Eastern Conference and Exhibition*, 1999.

[5] M. Schupack, "Corrosion Protection for Unbonded Tendons," *Concr. Int.*, vol. 13, no. 2, pp. 51–57, 1991.

[6] H. Ashar, C. P. Tan, and N. D, "Prestressed Concrete in U. S. Nuclear Power Plants (Part 1)," *Concr. Int.*, vol. 16, no. 5, pp. 30–34, 1994.

[7] J. L. Mongauze, "The VVER 1000 and 1200 prestressing system." Freyssinet, p. 49, 2016.

[8] E. Gallitre, P. Varpasuo, M. Sircar, and H. H. Abrishami, "Bonded or Unbonded Technologies for Nuclear Reactor Prestressed Concrete Containments," 2015.

[9] W. Podolny, "Corrosion of prestressing steels and its mitigation," *PCI J.*, pp. 34–55, 1992.

[10] J. Fuzier and A. Chabert, "Stay cable corrosion protection with petroleum wax," in *Extending the Lifespan of Structures [Vol. 2]*, 1995, pp. 891–896.

[11] J. Fuzier, H. Ganz, and P. Matt, "fib Bulletin 33: Durability of post-tensioning tendons," Lausanne, 2006.

[12] PTI, "Selection od Filler Material for Multistrand PT Tendons," Farmington Hills, 2015.

[13] C. W. Dolan, R. N. B. Jr, D. C. Buckner, N. H. Burns, M. Moore, H. R. Ganz, D. P. Jenny, S. N. Lane, D. H. Sanders, T. C. Schaeffer, M. Schupack, P. J. Sullivan, C. H. Walker, J. J. Zhao, K. H. Preston, and D. C. Pu, "Corrosion and Repair of Unbonded Single Strand Tendons - Committee 423," 1998.

[14] G. Fallis, C. Ball, and A. Moad, "Innovative Corrosion Evaluation System for Unbonded Post-Tensioned Cables," *Concr. Repair Bull.*, no. May/June, pp. 7–11, 2007.

[15] American Concrete Institute, "Corrosion of Prestressing Steels - Report 222.2R-01," Detroit, Michigan, 2001.

[16] H. Vander Vilde, "Condition evaluation and methods of corrosion protection for unbonded post tension cablesitle," 1999, p. Paper No. 9A.4.

[17] H. Ashar, C. P. Tan, and D. Naus, "Prestressed Concrete in U. S. Nuclear Power Plants (Part 2)," *Concr. Int.*, vol. 16, no. 6, pp. 58–61, 1994.

[18] B. Little and R. Ray, "Fungal Influenced Corrosion of Metals in Humid Environments," in *Corrosion and its control in the new millennium: Proceedings of the NACE International Northern Area Eastern Conference and Exhibition*, 1999.

[19] B. Little and R. Ray, "A Review of Fungal Influenced Corrosion," *Corros. Rev.*, vol. 19, p. 401, 2001.

[20] B. Little and R. Staehle, "Fungal influenced corrosion in post-tension structures," *Electrochem. Soc. Interface*, vol. 10, pp. 44–48, 2001.

[21] B. Little, R. Staehle, and R. Davis, "Fungal influenced corrosion of post-tensioned cables," *Int. Biodeterior. Biodegrad.*, vol. 47, no. 2, pp. 71–77, 2001.

[22] B. J. Little and R. I. Ray, "The Role of Fungi in Microbialogically Influenced Corrosion,"," in *15th International Corrosion Congress*, 2002.

[23] F. De Leo, G. Campanella, E. Proverbio, and C. Urzì, “Laboratory tests of fungal biocorrosion of unbonded lubricated post-tensioned tendons,” *Constr. Build. Mater.*, vol. 49, pp. 821–827, 2013.
[24] D. A. Canonico, J. C. Griess, and G. S. Robinson, “Final Report on PCRV Thermal Cylinder Axial Tendon Failures,” Oak Ridge, Tennesse, 1976.
[25] J. F. Costello, D. J. Naus, and C. B. Oland, “An Investigation of Tendon Corrosion-Inhibitor Leakage into Concrete,” in *Expertcentrum, International conference*, 1999, pp. 122–127.
[26] J. C. Griess and D. J. Naus, “Corrosion of Steel Tendons Used in Prestressed Concrete Pressure Vessels,” in *Corrosion of Reinforcing Steel in Concrete, ASTM STP 713*, 1980, pp. 32–50.
[27] NRC, “Joint Petitioners’ supplemental basis for previously submitted contention 6-TVA has not and can not meet the NRC’s quality assurance adn quality control requirements.” Bellefonte Nuclear Power Plant, Tennessee, 2010.
[28] D. A. Copinger and D. L. Moses, “Fort Saint Vrain Gas Cooled Reactor Operational Experience,” Oak Ridge, Tennesse, 2004.
[29] Fédération internationale du béton, *fib Bulletin 30: Acceptance of stay cable systems using prestressing steels*. Lausanne: F´ed´eration Internationale du B´eton, 2005.
[30] E. ETAG-013, “Guideline for European Technical Approval of Post-Tensioning Kits for Prestressing of Structures,” 2002.
[31] J. Mietz, A. Burkert, A. Burkert, and G. Eich, “Investigations on the protection effect of filling materials for post-tensioning systems under construction conditions,” *Mater. Corros.*, vol. 57, no. 11, pp. 843–851, 2006.
[32] Sanchem, “Hot Application Rust Preventatives, No-Ox-ID,” 2016. [Online]. Available: http://www.sanchem.com/docs/NO-OX-ID A.pdf.
[33] Lubricant_Specialities_Company, “Red i PT Cable Coating Grease,” 2010. [Online]. Available: http://www.lsc-online.com/red-i-pt-cable-coating-grease/.
[34] Lubebase, “Specis B 4851-2,” 2008. [Online]. Available: http://lubebase.com/cgi-bin/ctl/lube/Total-SPECIS-B-4851-2?id=5043.
[35] Sonneborn, “Visconorust 2090 P-4,” *Sonneborn, Refined Products*, 2013. [Online]. Available: http://www.sonneborn.com.
[36] Fuchs, “Industrial-Corrosion Preventives - Anticorit 28890,” 2016. [Online]. Available: https://www.fuchs.com/ca/en/brands/a-k/anticorit/.
[37] B. Godart, “Status of durability of post-tensioned tendons in France,” in *Durability of post-tensioning tendons. Proceedings of Workshop, 15-16 November, Ghent (Belgium)*, 2001, vol. Bulletin 1, pp. 25–42.
[38] M. Virlogeux, “External prestressing,” in *Developments in prestressed concrete structures: part I*, 1982, pp. 101–108.
[39] M. Traute and H. Weiher, “Improved sustainability in post-tensioning by replaceable internal unbonded tendons,” *fib Symp. PRAGUE 2011 Concr. Eng. Excell. Effic. Proc.*, vol. 2, no. February, pp. 831–834, 2011.
[40] R. Woodward, “Durability of Post-Tensioned Tendons on Road Bridges in the UK,” [International Federation for Structural Concrete], [Lausanne], 2009.
[41] K. Zilch, H. Weiher, and M. Riesemann, “Concrete Bridges in Germany,” in *7th German-Japanese Bridge Symposium*, 2007.
[42] H. Weiher, H. Thal, and K. und Zilch, “Verbundlose Vorspannung im Brü-cken- und Ingenieurbau, Eigenveröf-fentlichung der Fa. VBT GmbH Thal-gau-Austria, www.vbt-

systems.eu."
[43] H. Weiher, "Verhalten von PE-HD Schutzhüllen bei dur Umlenkung von verbundlosen Spanngliedern," *MÜNCHEN*, 2007.
[44] I. P. Gilles, "Internal post-tensioning tendons: many problems and sometimes solutions," in *17e Colloque sur la progression de la recherche québécoise sur les ouvrages d'art*, 2010, pp. 1–10.
[45] R. M. Ebeling, R. W. Strom, J. E. H. Jr, R. W. Haskins, and J. A. Evans, "Assessing Corrosion Damage and Corrosion Progression in Multistrand Anchor Systems in Use at Corps Projects Engineer Research and Development," Vicksburg, MS, 2013.
[46] H. Brooman and A. Robson, "A3/A31 Flyover - Case History of an Externally Post-Tensioned Bridge," in *Proceeding of the Seventh International Conference on Structural Faults and Repair*, 1997, pp. 307–315.
[47] B. J. Little, T. L. Gerke, R. I. Ray, and J. S. Lee, "Chapter 5 - The Mineralogy of Microbiologically Influenced Corrosion," Z. Amjad and K. D. B. T.-M. S. and D. Demadis, Eds. Amsterdam: Elsevier, 2015, pp. 107–122.
[48] K. C. Marshall, "Advances in Microbial Ecology. Chapter 6: The Ecology of Microbial Corrosion," *Kensington, New South Wales*, vol. 11, pp. 240–242, 1990.
[49] E. G. Toropova, A. A. Gerasimenko, and A. A. Gureev, "Biological damage to lubricants," *Chem Technol Fuels Oils*, vol. 24, no. 11, pp. 489–491, 1988.
[50] A. Lugauskas, I. Prosyčevas, R. Ramanauskas, A. Grigucevičiene, A. Selskiene, and V. Pakštas, "The Influence of micromycetes on the corrosion behaviour of metals (steel, al) under conditions of the environment polluted with organic substances," *Medziagotyra*, vol. 15, no. 3, pp. 224–235, 2009.
[51] O. P. Savitskaya, "Investigation of Carbonyl Corrosion in Metals," *Chem. Pet. Eng.*, vol. 8, pp. 604–605, 1965.
[52] M. Peter, "Inspection and Maintenance of Post-Tension Tendons," Zurich, 1987.
[53] D. W. Cullington, "Non-Destructive Methods for Inspection of Post-Tensioned Concrete Bridges," in *FIB Symposium on Post-Tensioned Concrete Structures*.
[54] Trenton, "Trenton Products," *Trenton Corporation*, 2014. [Online]. Available: http://trentoncorp.com/wp-content/uploads/2016/01/20005e_trenton_corp_fill-coat2_rev0.pdf.
[55] M. Vill, E. M. Eichinger, and J. Kollegger, "Assessment of Damaged Post-Tensioning Tendons," *Struct. Eng. Int.*, vol. 16, no. 1, pp. 44–48, Feb. 2006.
[56] Fédération internationale du béton, "Durability of post-tensioning tendons," Ghent, Belgium, 2001.
[57] C. Waalwijk, J. R. A. de Koning, R. P. Baayen, and W. Gams, "Discordant Groupings of Fusarium spp. from Sections Elegans, Liseola and Dlaminia Based on Ribosomal ITS1 and ITS2 Sequences," *Mycologia*, vol. 88, no. 3, pp. 361–368, 1996.
[58] M. S. El-Abyad, A. M. Abu-Taleb, and M. Ghareeb, "The effects of Fusarium oxysporum f.sp. lycopersici (Sacc.) Snyder & Hansen on tomato in diphenamid-treated soil," *Mycopathologia*, vol. 119, no. 1, pp. 35–41, 1992.
[59] L. R. Gale, T. Katan, and H. C. Kistler, "The probable center of origin of Fusarium oxysporum f. sp lycopersici VCG 0033," *Plant Dis.*, vol. 87, no. 12, pp. 1433–1438, 2003.
[60] R. A. Samson, J. Houbraken, U. Thrane, J. C. Frisvad, and B. Andersen, "Food and Indoor Fungi ," vol. 2. CBS-KNAW Fungal Biodiversity Centre , 2010.
[61] L. A. Ramírez-Camejo, A. Zuluaga-Montero, M. Lázaro-Escudero, V. Hernández-Kendall,

and P. Bayman, “Phylogeography of the cosmopolitan fungus Aspergillus flavus: Is everything everywhere?,” *Fungal Biol.*, vol. 116, no. 3, pp. 452–463, 2012.
[62] U. L. Diener, R. J. Cole, T. H. Sanders, G. A. Payne, L. S. Lee, and M. A. Klich, “Epidemiology of Aflatoxin Formation by Aspergillus Flavus*,” *Annu. Rev. Phytopathol.*, vol. 25, no. 1, pp. 249–270, 1987.
[63] H. R. Hamilton and J. A. Rice, *Replaceable Unbonded Tendons for Post-Tensioned Bridges (Task 1)*, no. 000112216. Gainesville, FL, 2015.

Appendices

Appendix A

Figure 160. View of strand wires coated with Civetea after removing filler and cleaning

Figure 161. View of wire 1 after removing filler and cleaning

Figure 162. View of wire 2 after removing filler and cleaning (full view).

Figure 163. View of wire 3 after removing filler and cleaning (full view).

Figure 164. Close-up of wire 4 after removing filler and cleaning (view 1)

Figure 165. Close-up of wire 5 after removing filler and cleaning (view 2).

Figure 166. Close-up of wire 6 after removing filler and cleaning (view 2)

Figure 167. Close-up of wire 7 after removing filler and cleaning

Figure 168. View of second strand segment: wires after removing filler and cleaning

Figure 169. View of 2nd strand segment, wire 1 after removing filler and cleaning

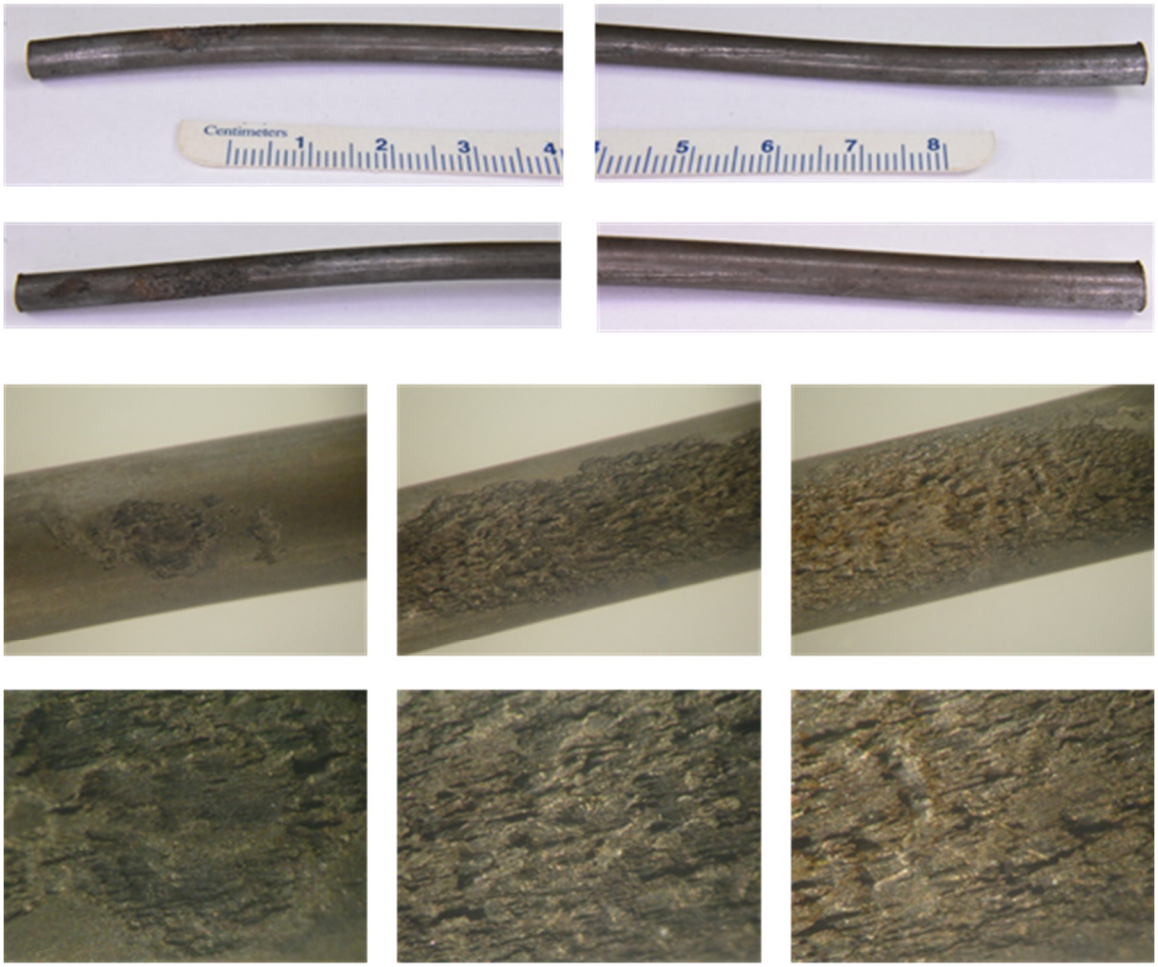


Figure 170. View of 2nd strand segment, wire 2 after removing filler and cleaning

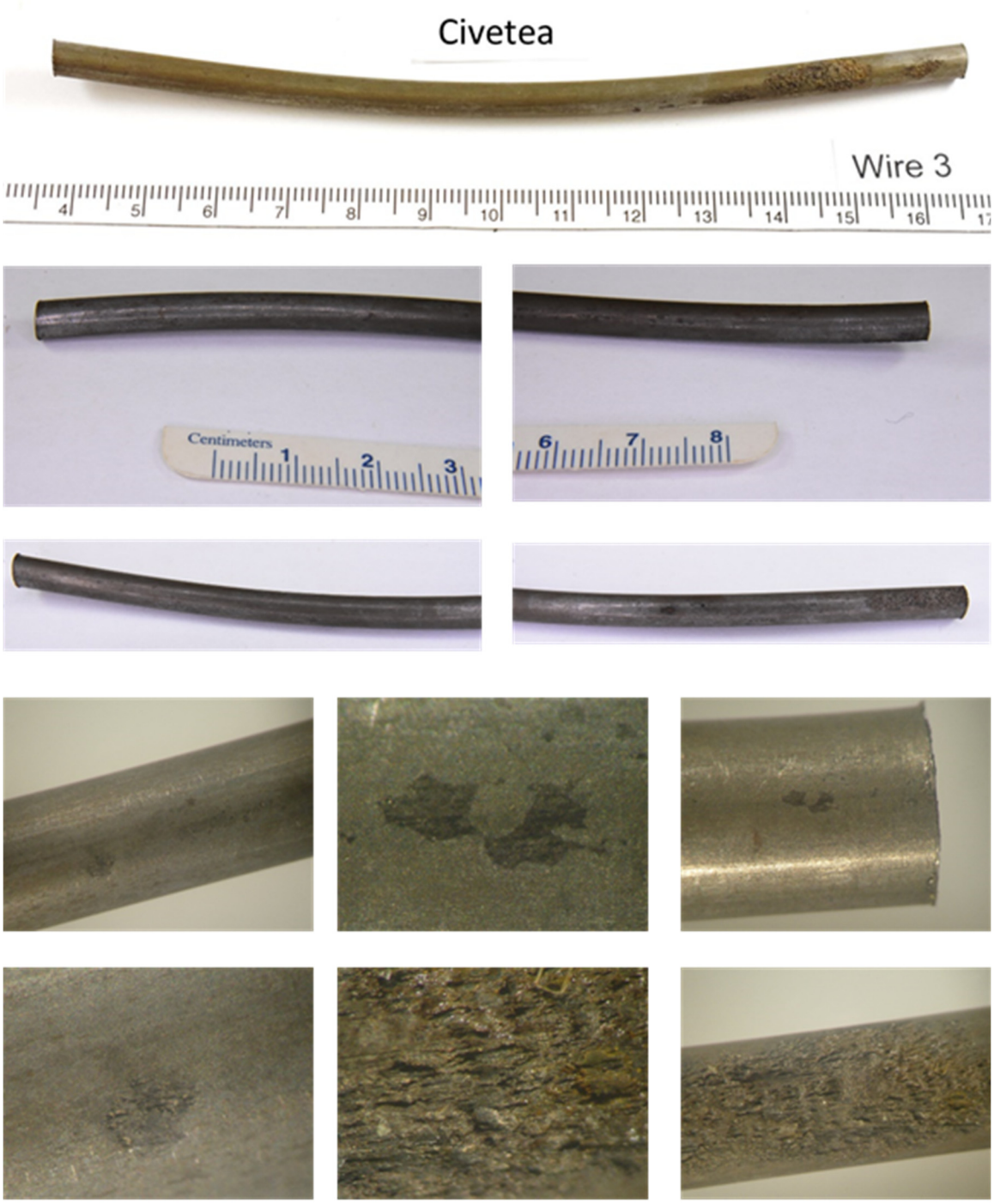


Figure 171. View of 2nd strand segment, wire 3 after removing filler and cleaning

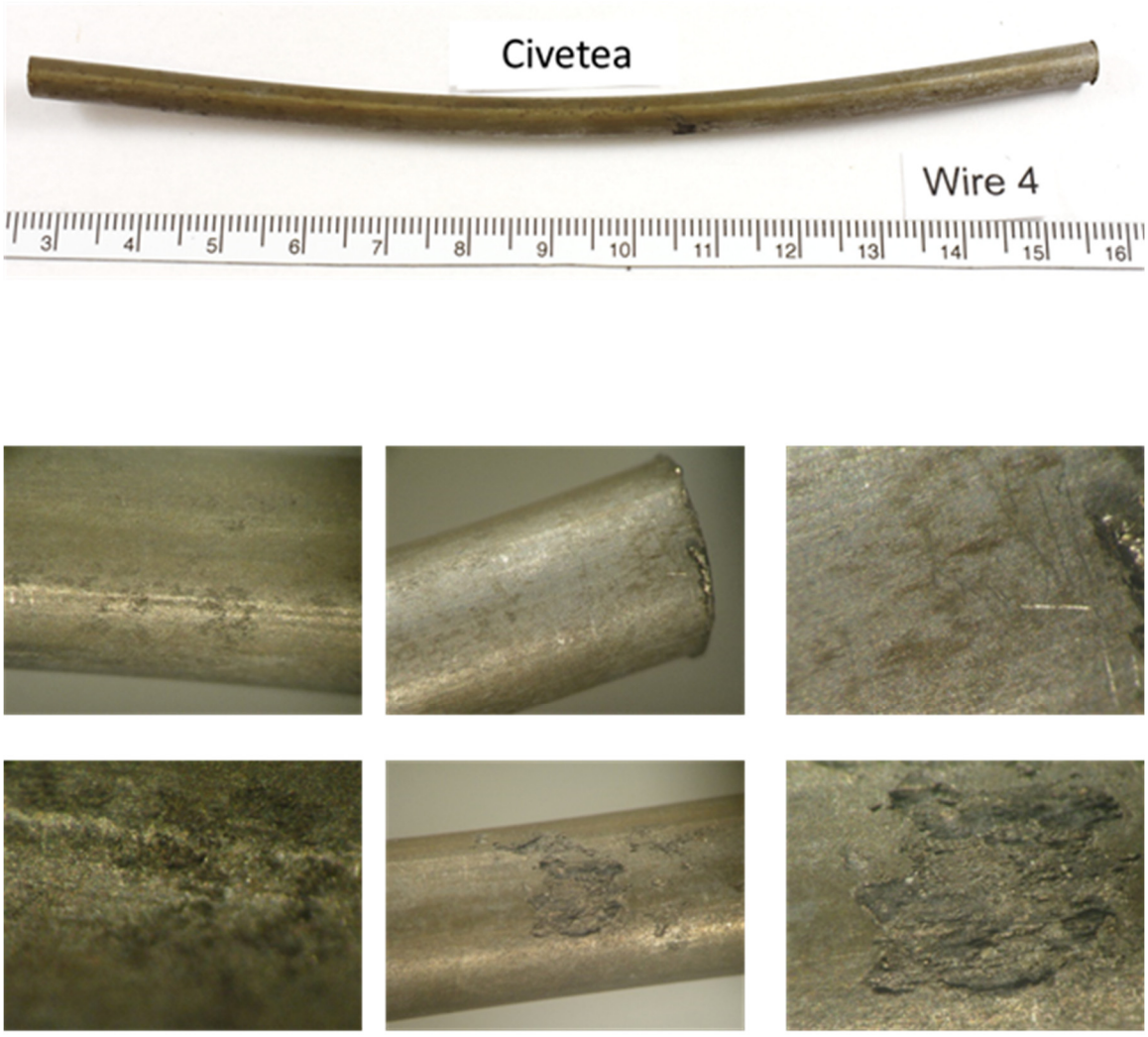


Figure 172. View of 2nd strand segment, wire 4 after removing filler and cleaning

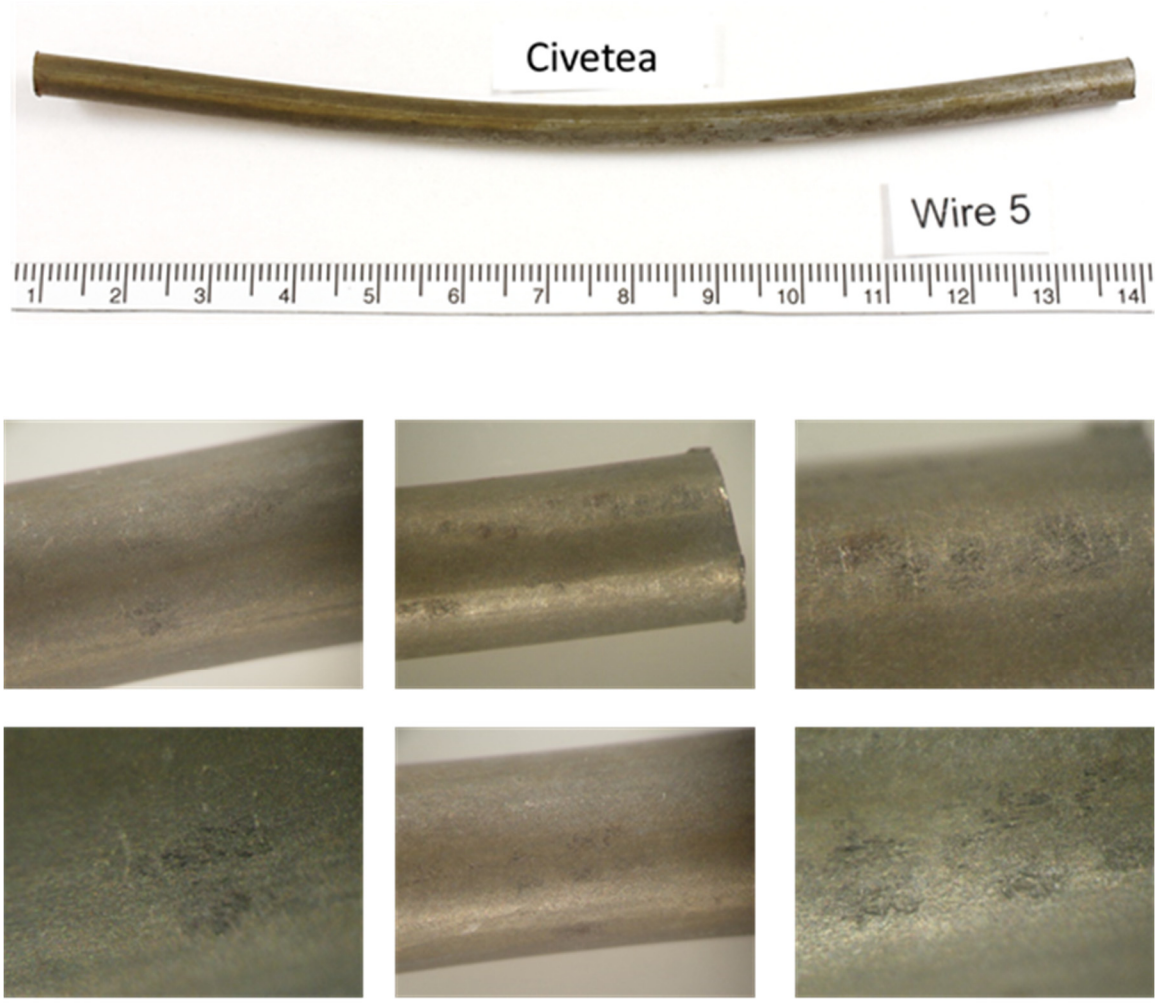


Figure 173. View of 2nd strand segment, wire 5 after removing filler and cleaning

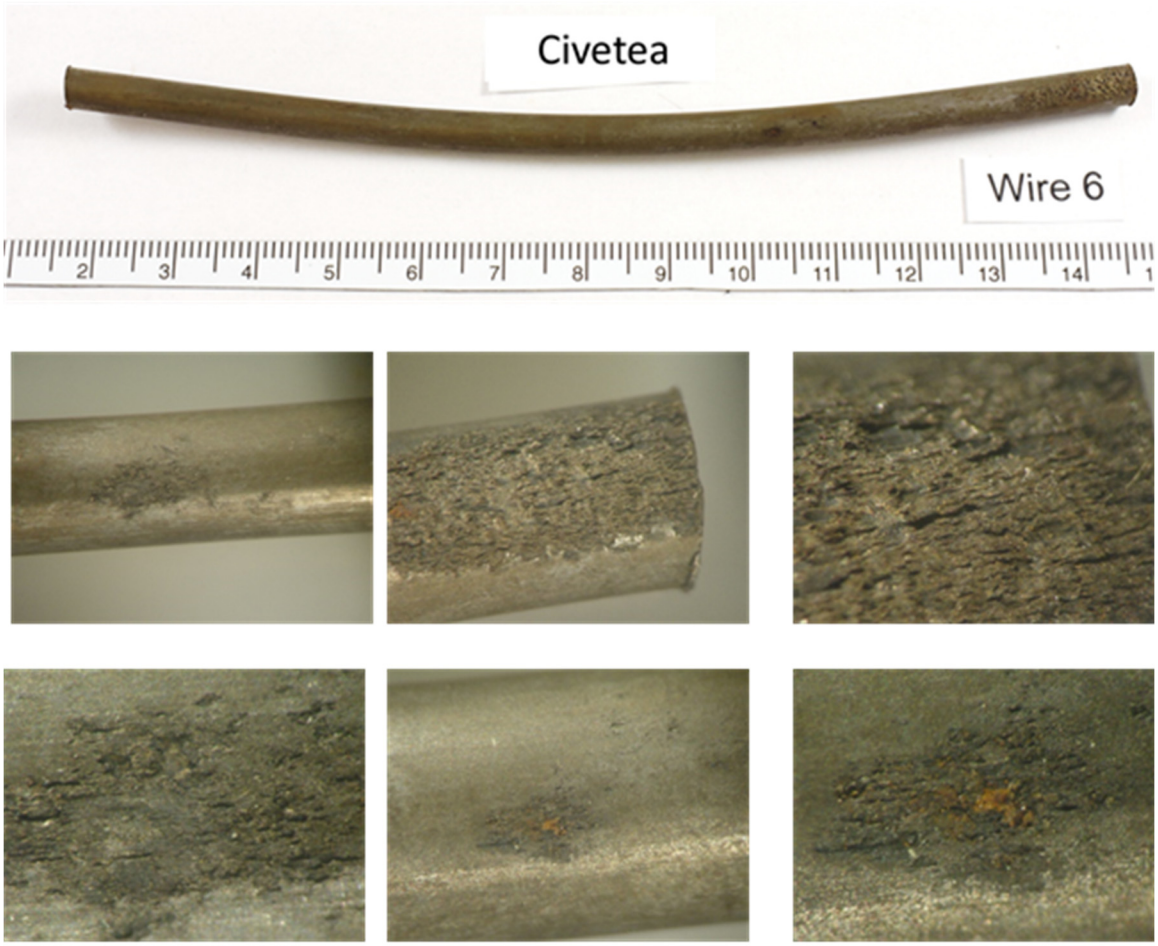


Figure 174. View of 2nd strand segment, wire 6 after removing filler and cleaning

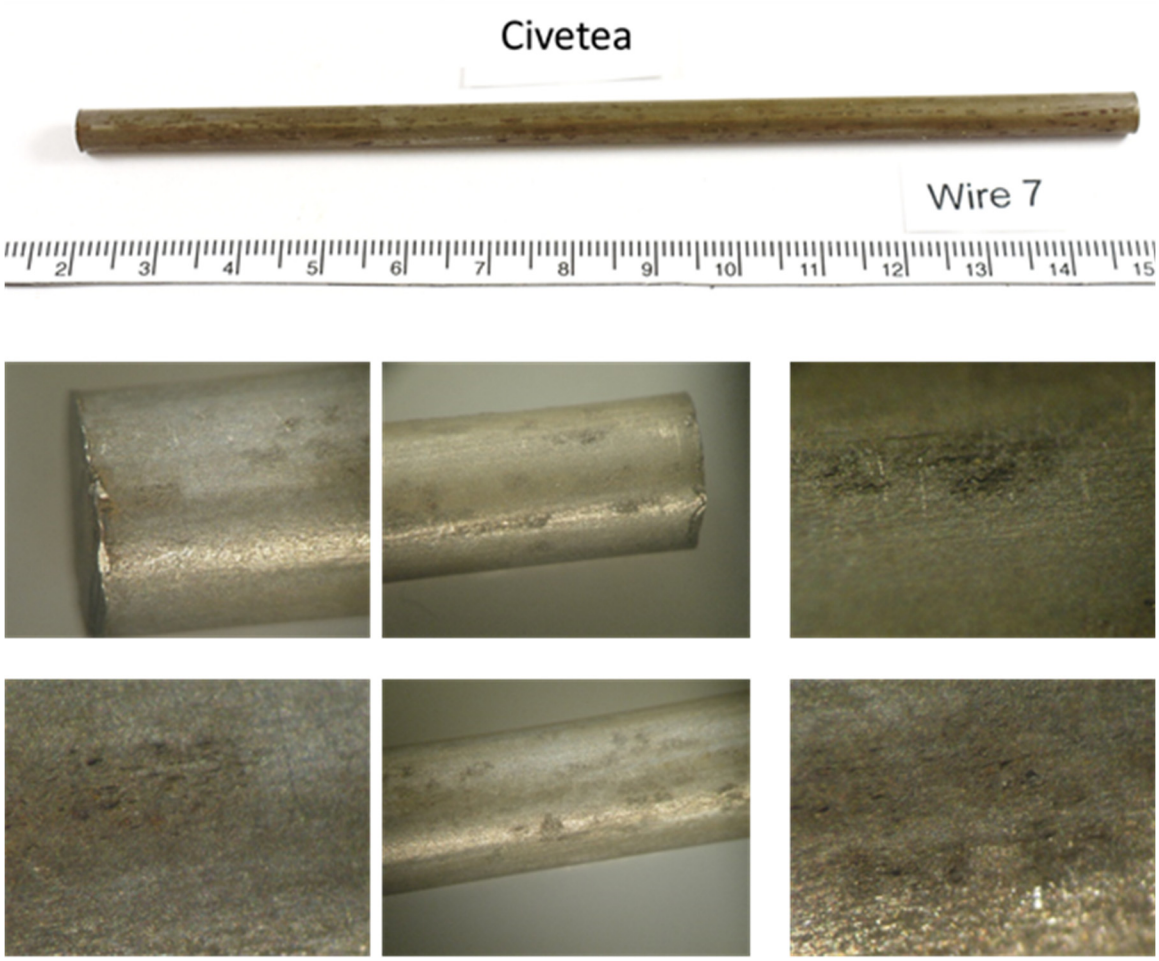


Figure 175. View of 2nd strand segment, wire 7 after removing filler and cleaning

Figure 176. View of strand wires after removing Nontribos filler and cleaning (edge piece)

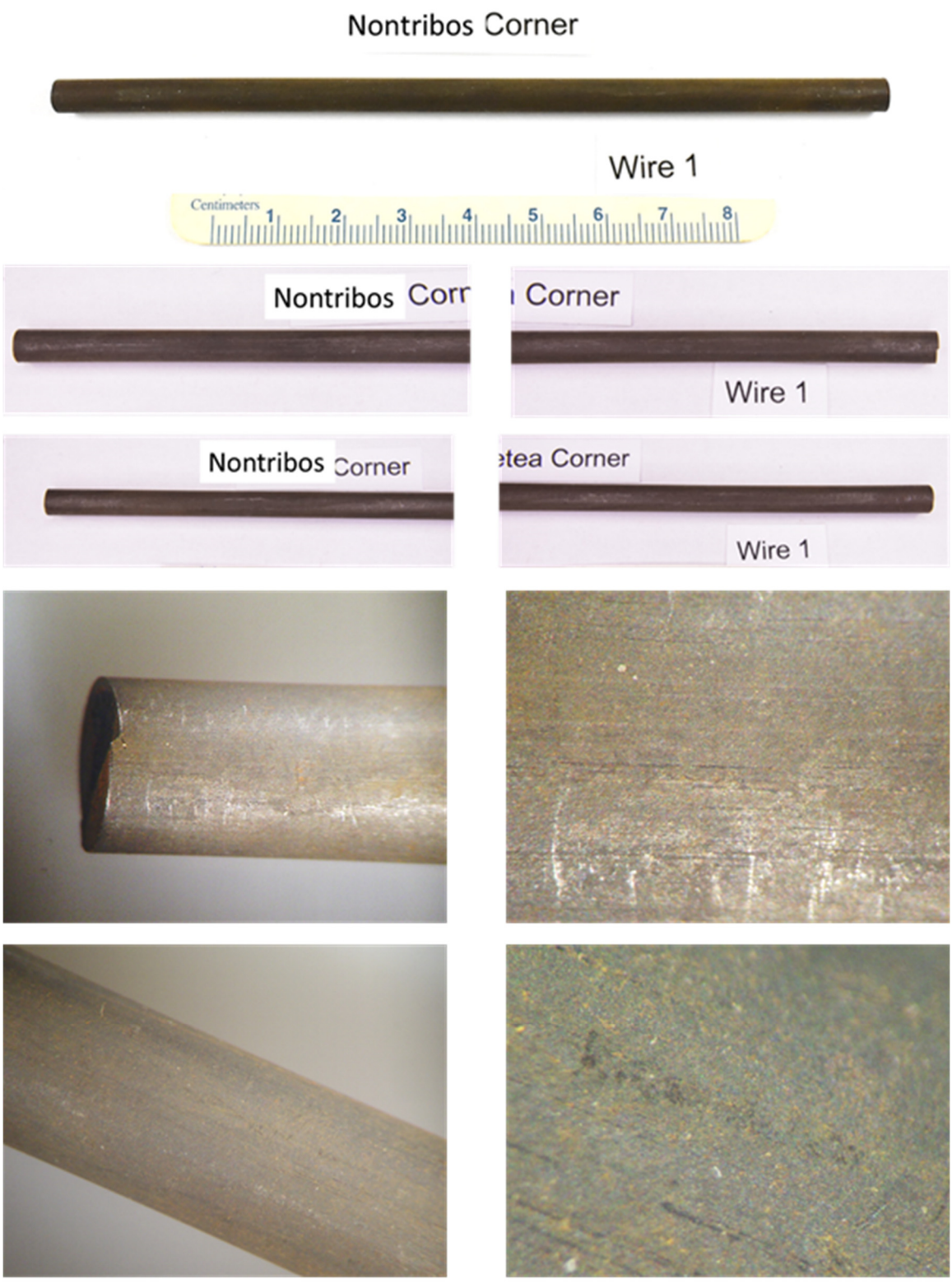


Figure 177. View of 1st strand segment, wire 1 after removing filler and cleaning (close-up 2)

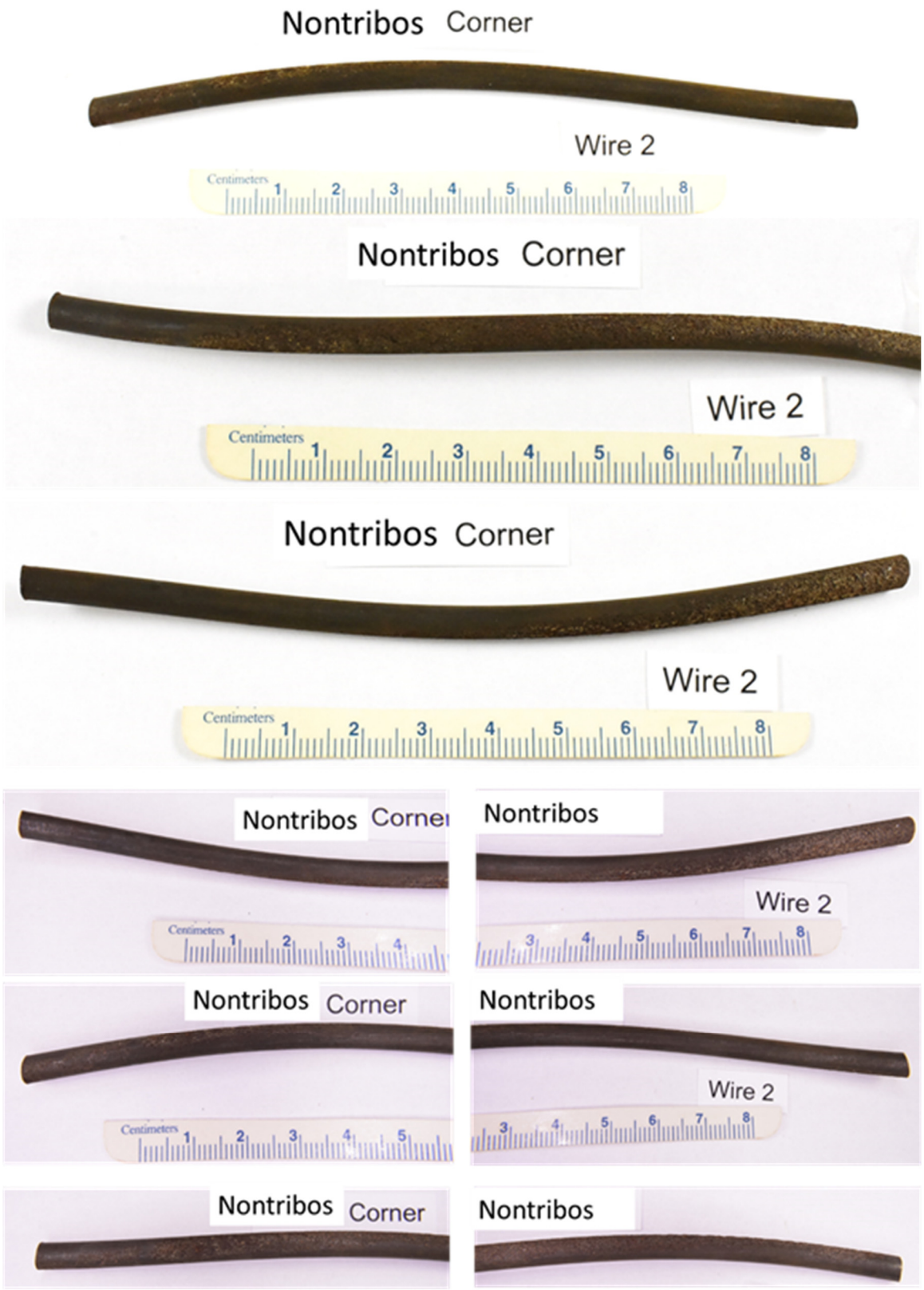


Figure 178. View of 1st strand segment, wire 2 after removing filler and cleaning (close-up 3)

Figure 179. View of 1st strand segment, wire 2 after removing filler and cleaning (close-up 3)

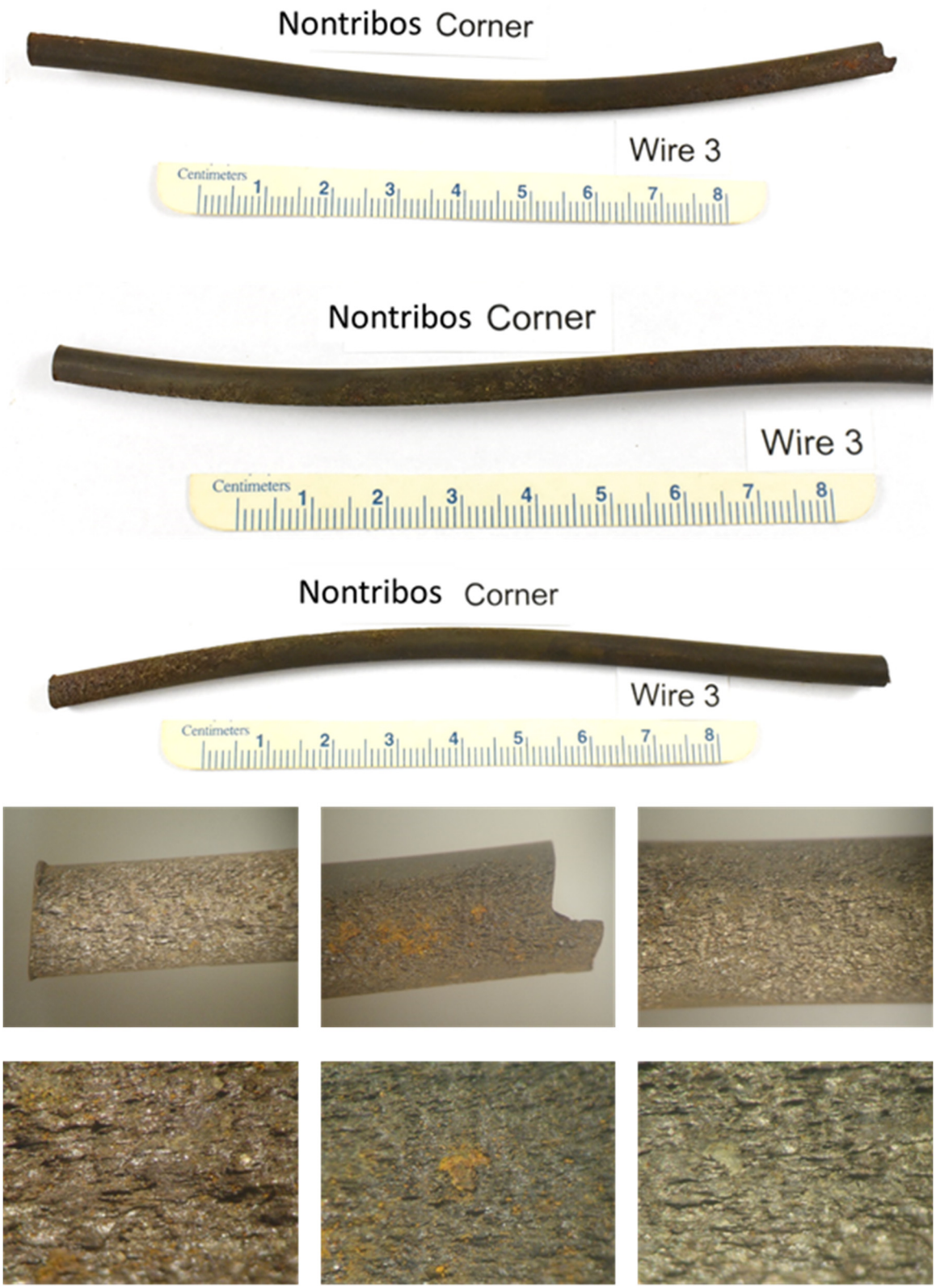


Figure 180. View of 1st strand segment, wire 3 after removing filler and cleaning (close-up)

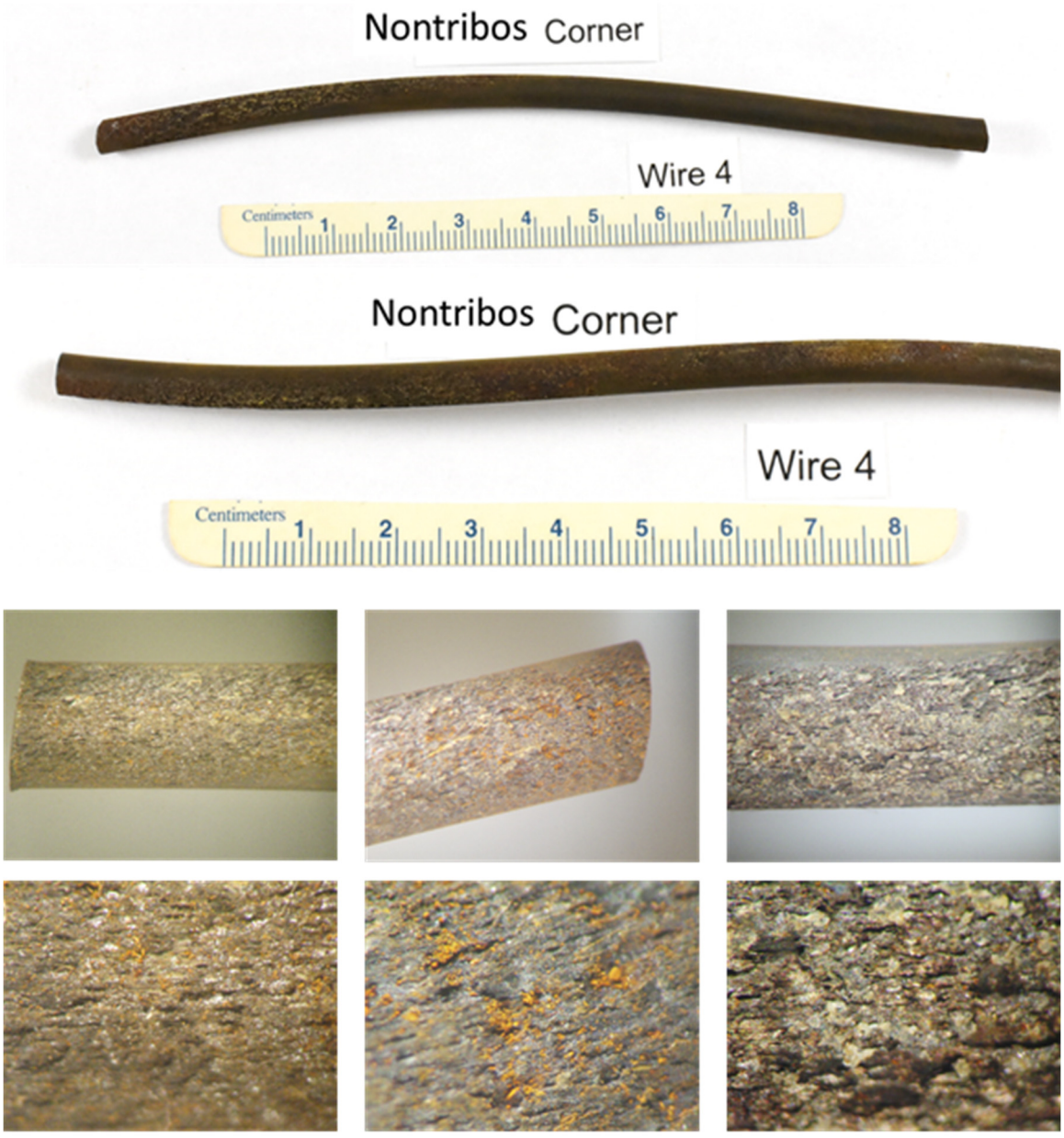


Figure 181. View of 1st strand segment, wire 4 after removing filler and cleaning

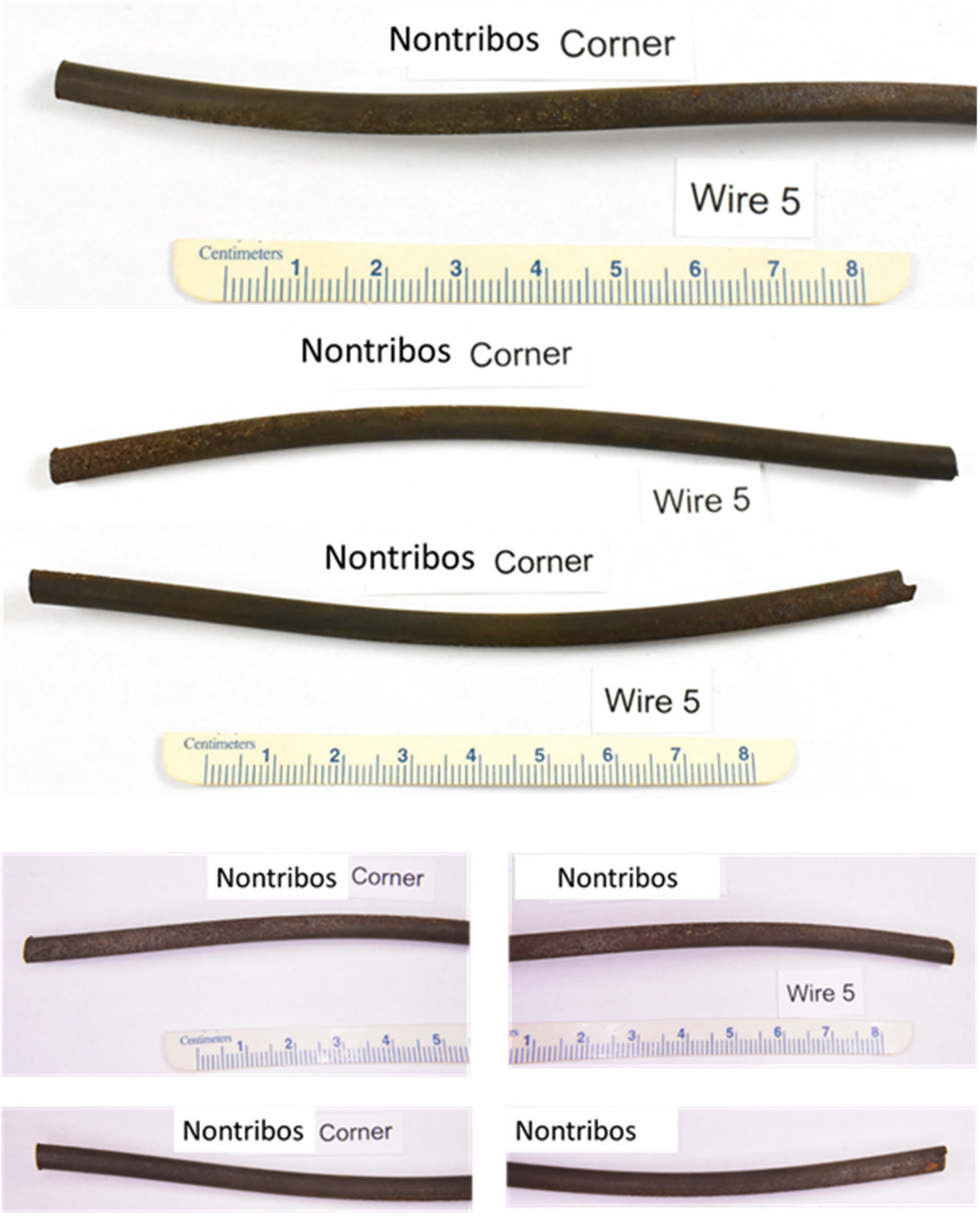


Figure 182. View of 1st strand segment, wire 5 after removing filler and cleaning

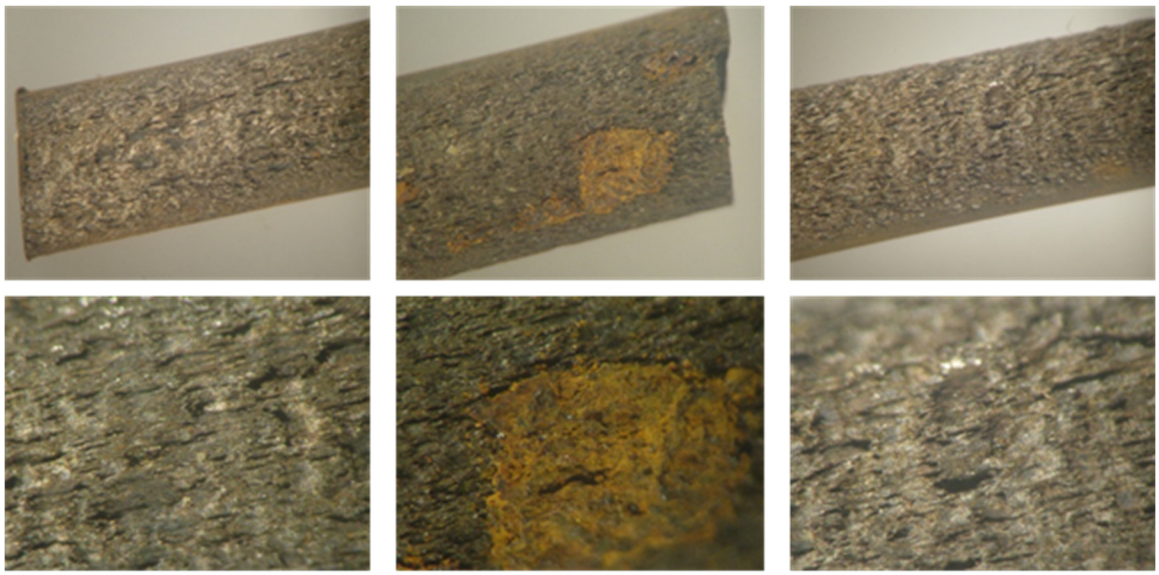


Figure 183. View of 1st strand segment, wire 5 after removing filler and cleaning (close-up).

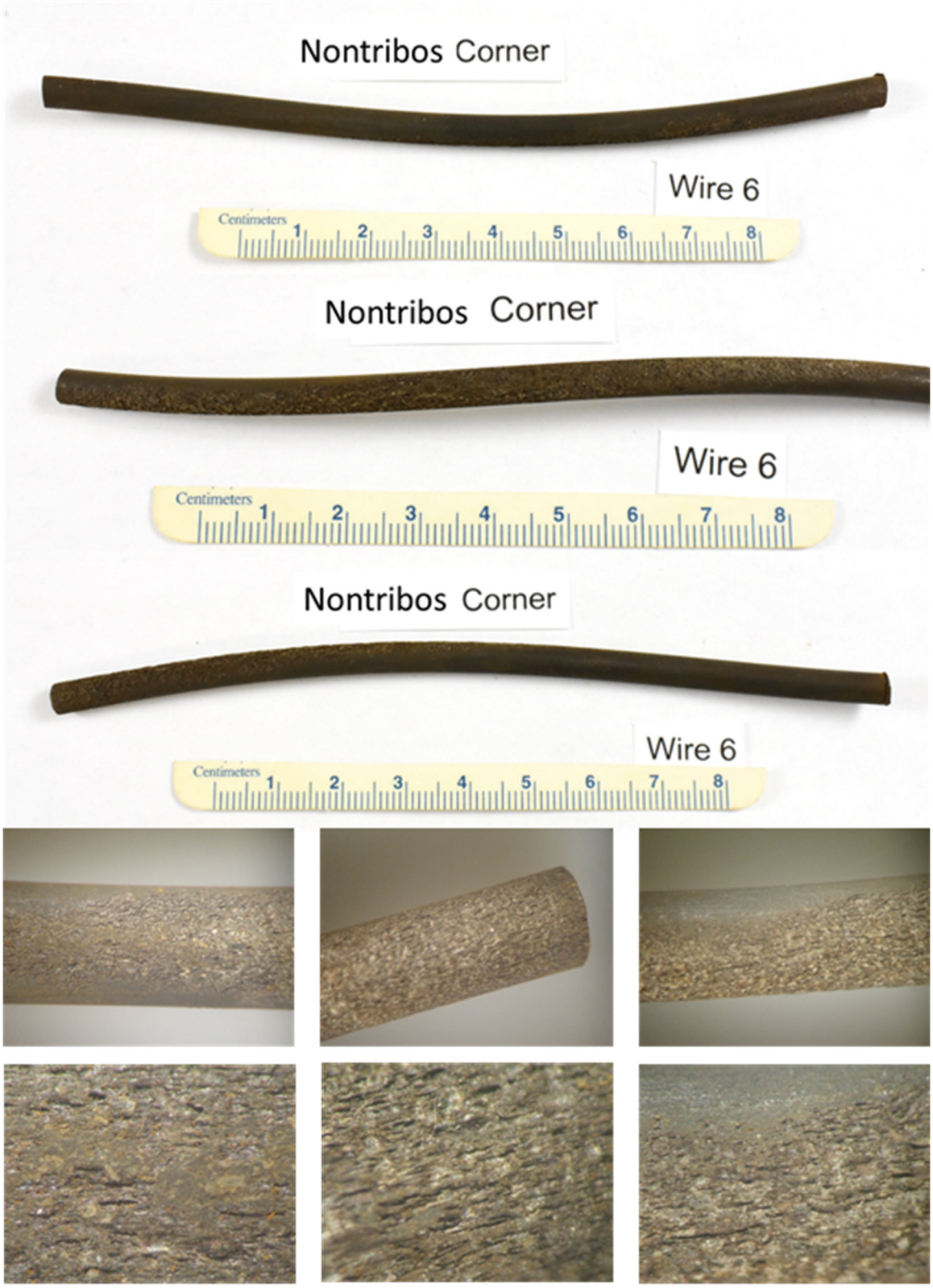


Figure 184. View of 1st strand segment, wire 6 after removing filler and cleaning. (close-up).

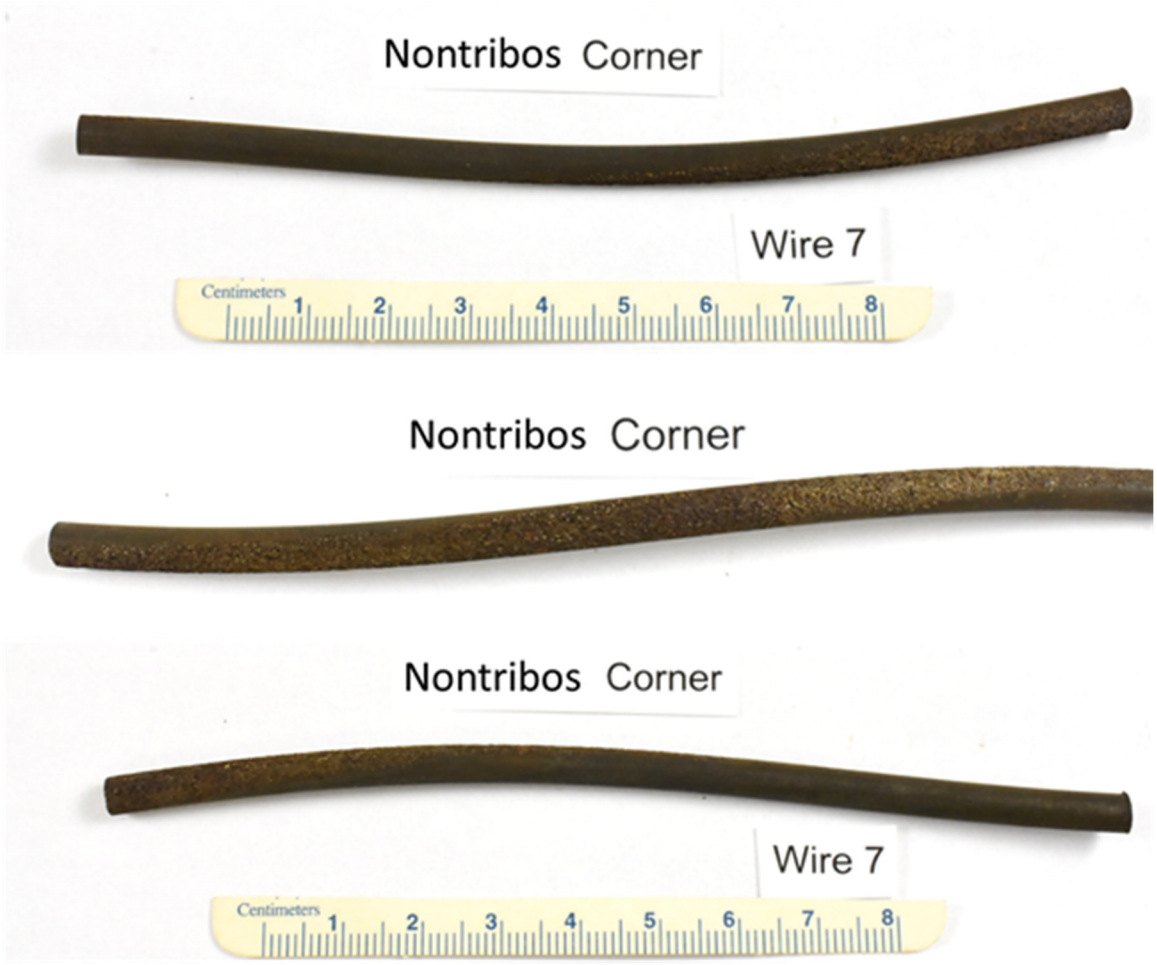


Figure 185. View of 1st strand segment, wire 7 after removing filler and cleaning. (close-up)

Figure 186. View of strand wires after removing Nontribos filler and cleaning

Figure 187. View of 2nd strand segment, wire 1 after removing filler and cleaning

Figure 188. View of 2nd strand segment, wire 2 after removing filler and cleaning (close-up)

Figure 189. View of 2nd strand segment, wire 3 after removing filler and cleaning (close-up)

Figure 190. View of 2nd strand segment, wire 4 after removing filler and cleaning (close-up)

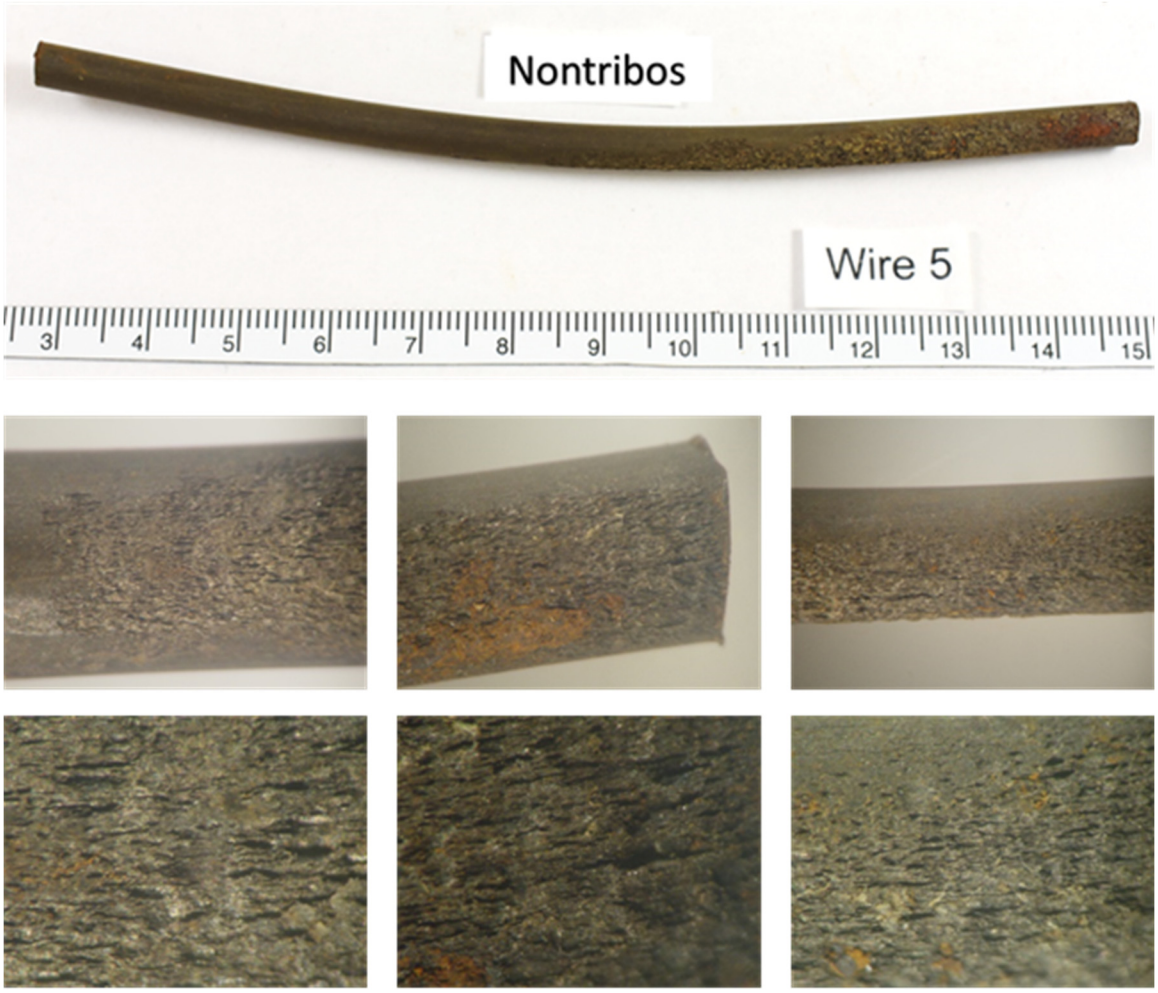


Figure 191. View of 2nd strand segment, wire 4 after removing filler and cleaning. (close-up 3).

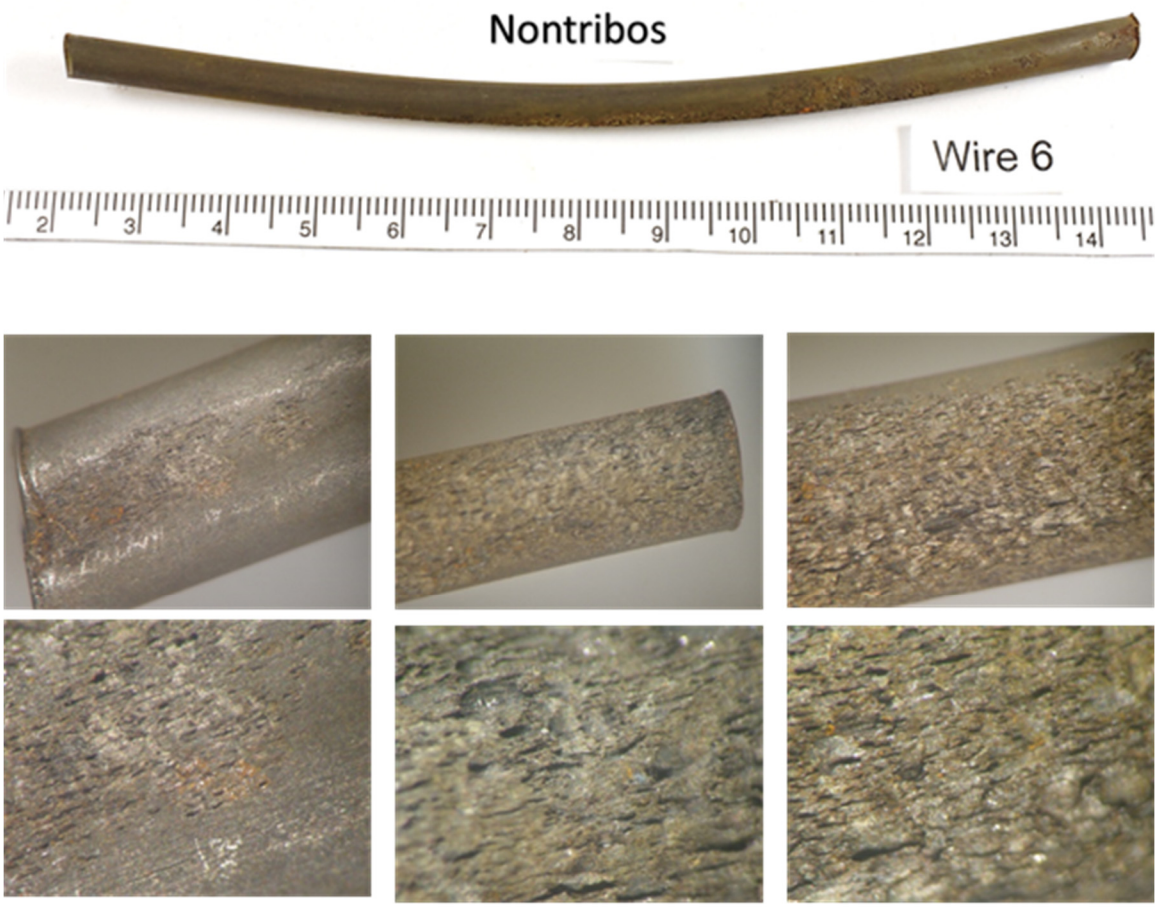


Figure 192. View of 2nd strand segment, wire 6 after removing filler and cleaning (close-up 1)

Figure 193. View of 2nd strand segment, wire 7 after removing filler and cleaning (close-up 1)

Figure 194. View of coated strand with Trenton (outside) edge (view 1).

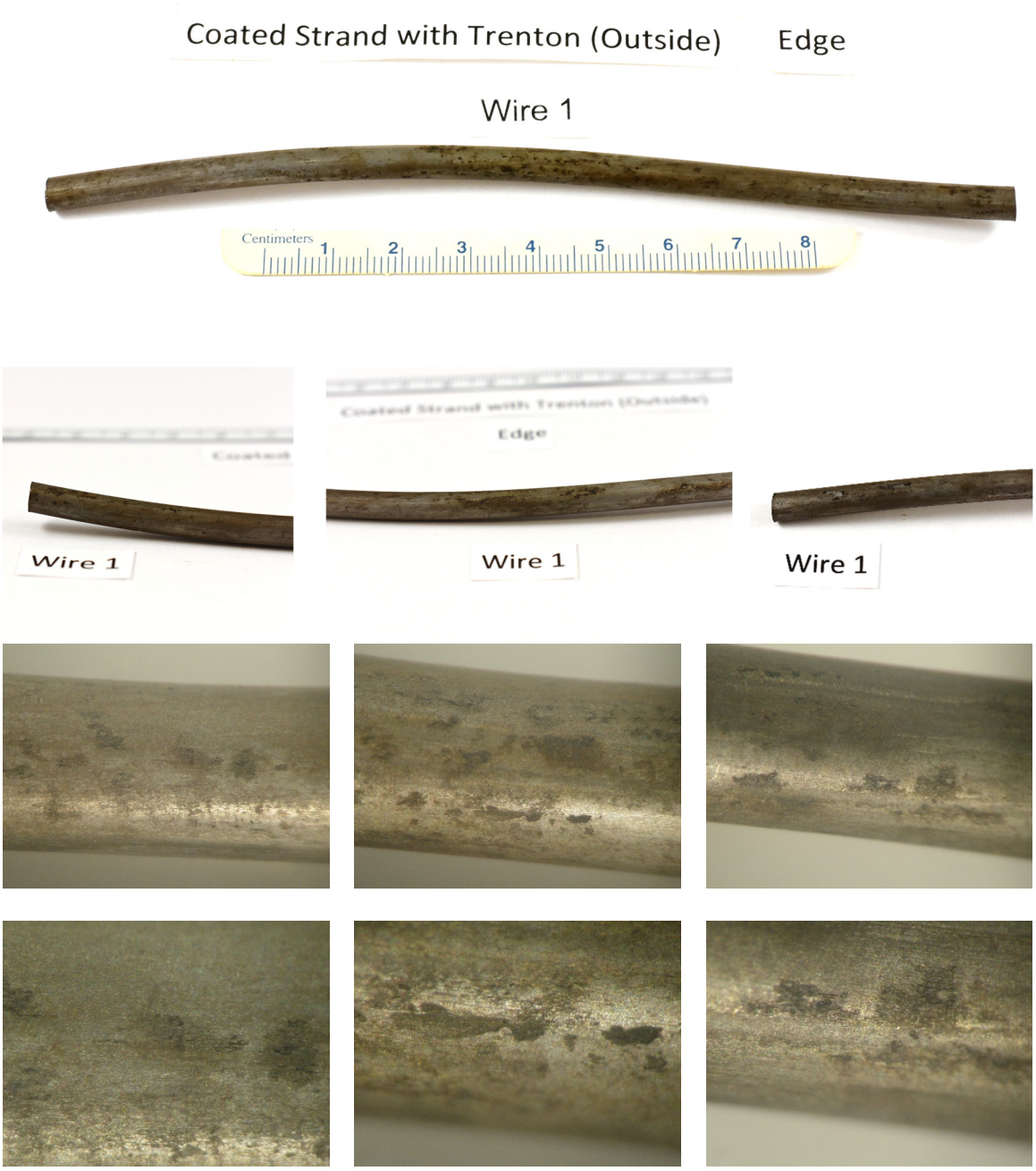


Figure 195. View of coated strand with Trenton (outside) edge, wire 1 after removing filler and cleaning (close-up)

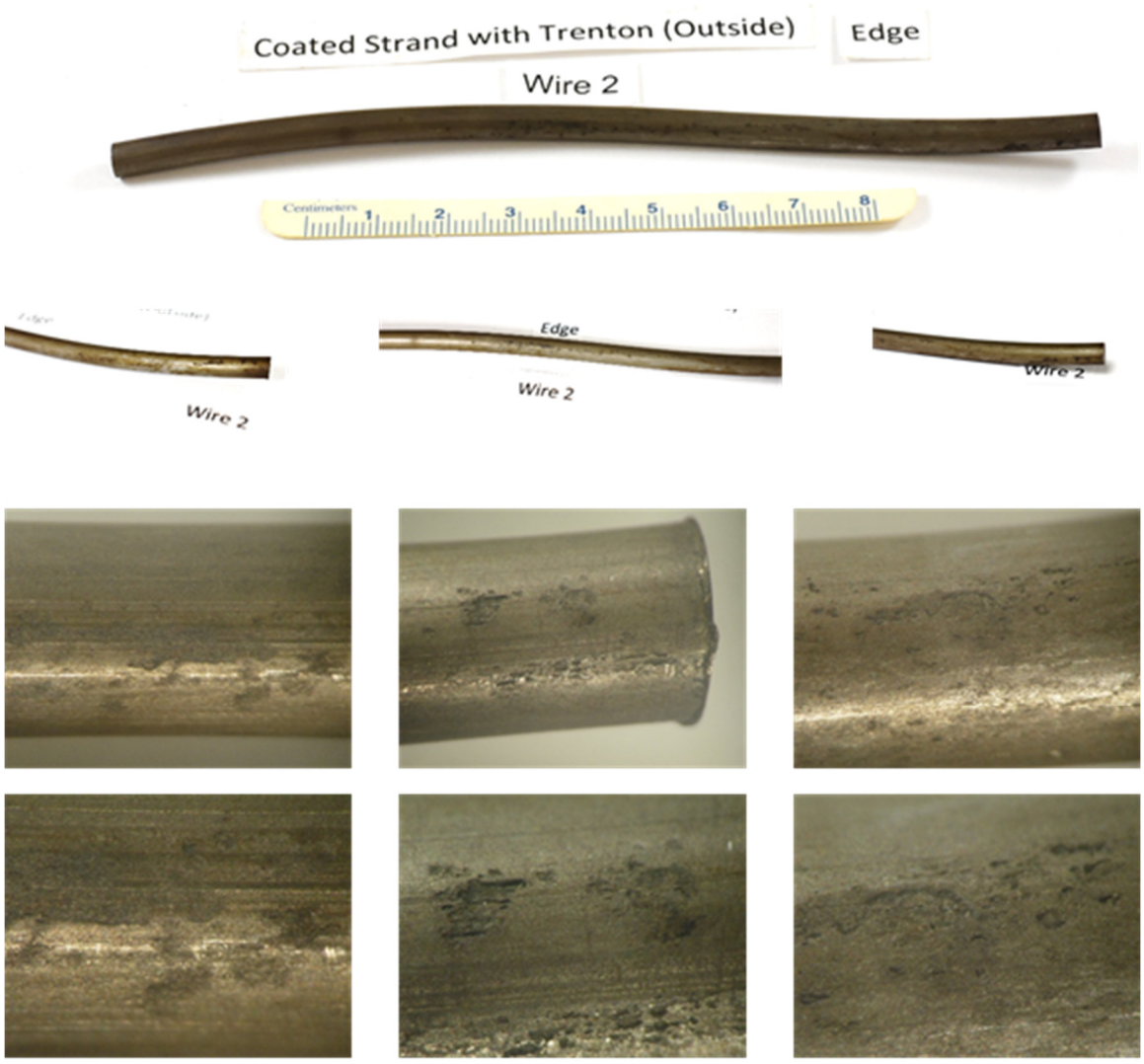


Figure 196. View of coated strand with Trenton (outside) edge, wire 2 after removing filler and cleaning (close-up 2)

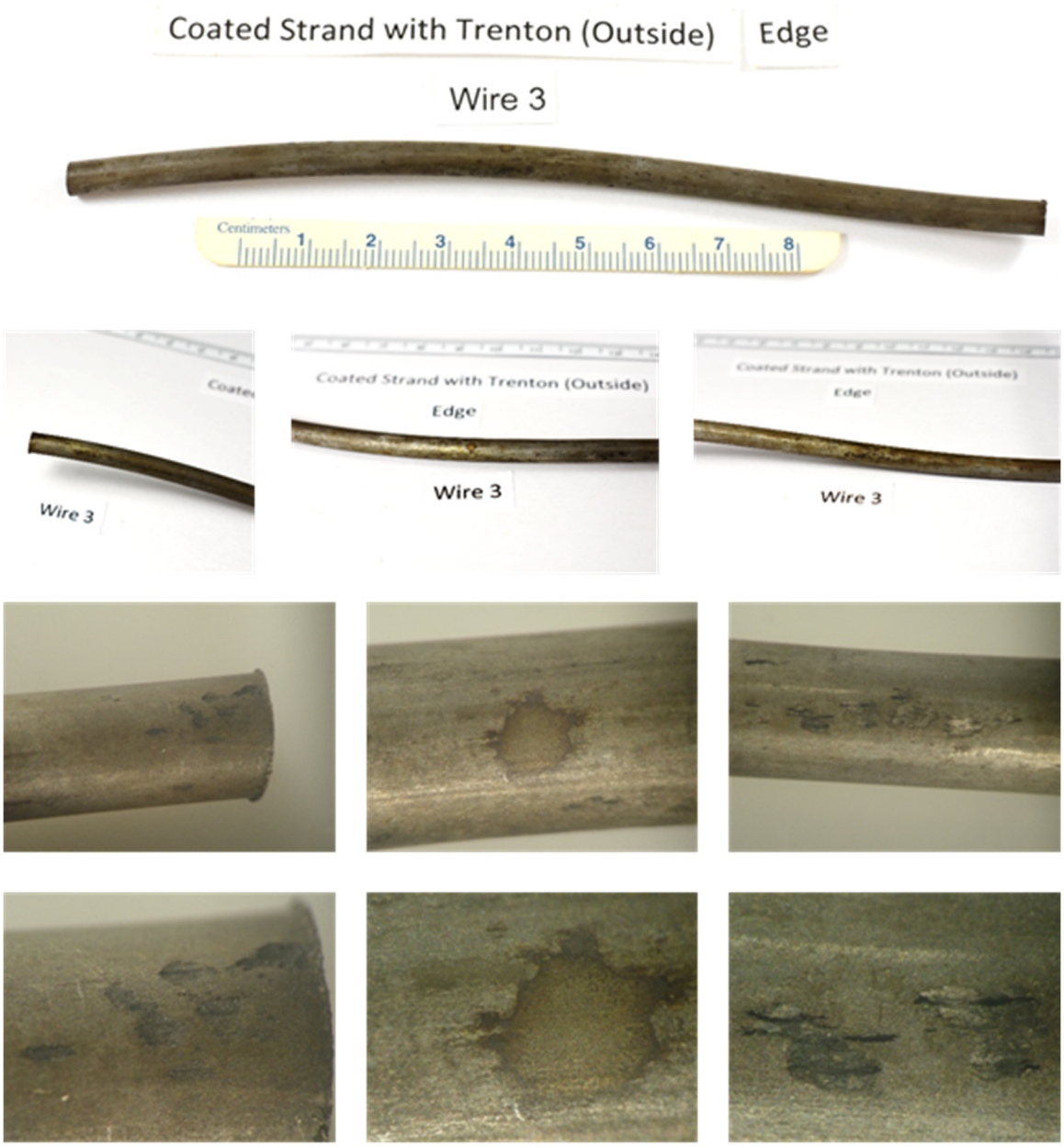


Figure 197. View of coated strand with Trenton (outside) edge, wire 3 after removing filler and cleaning (close-up 2)

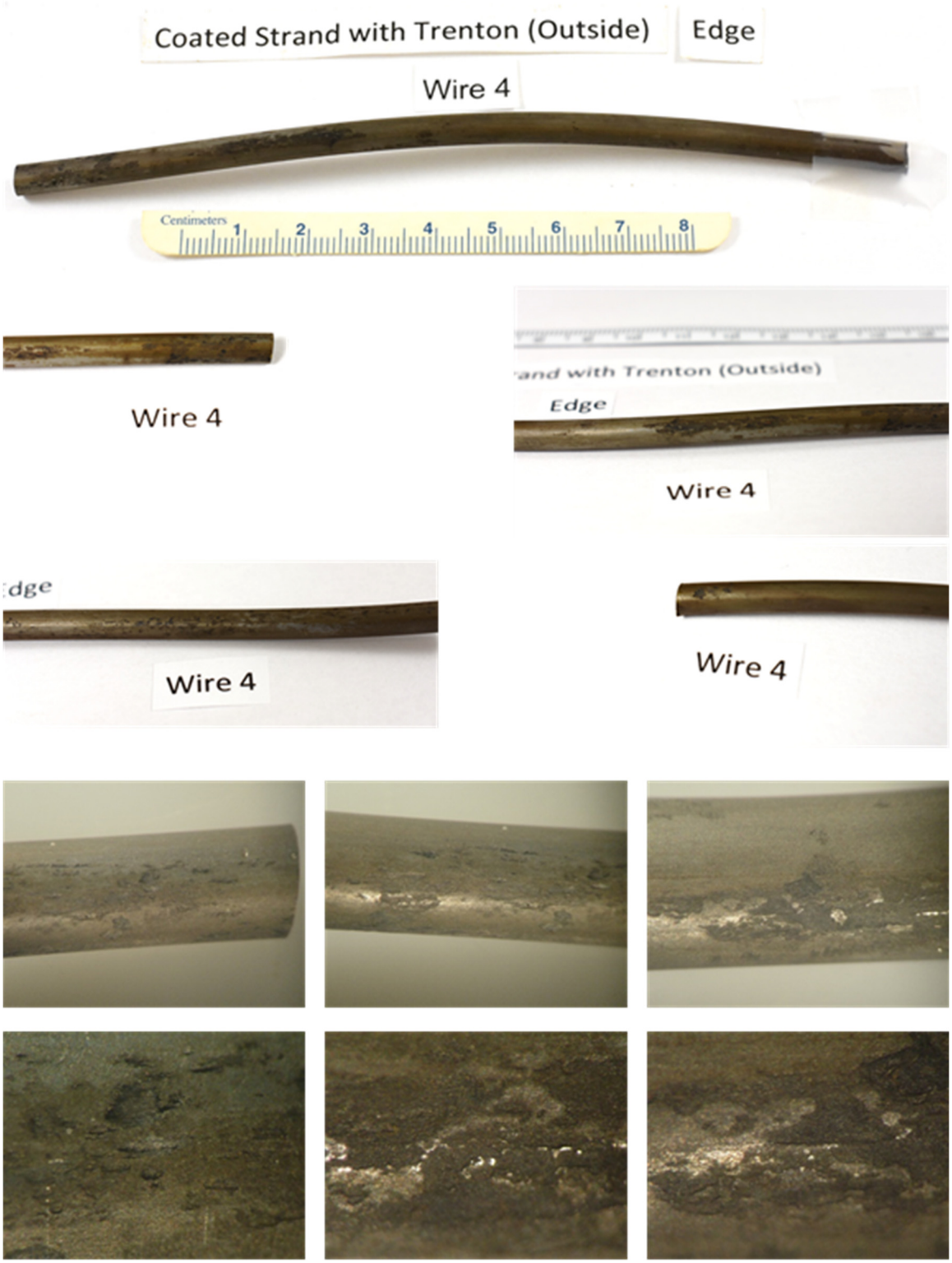


Figure 198. View of coated strand with Trenton (outside) edge, wire 4 after removing filler and cleaning (close-up 2)

Figure 199. View of coated strand with Trenton (outside) edge, wire 5 after removing filler and cleaning (close-up 2)

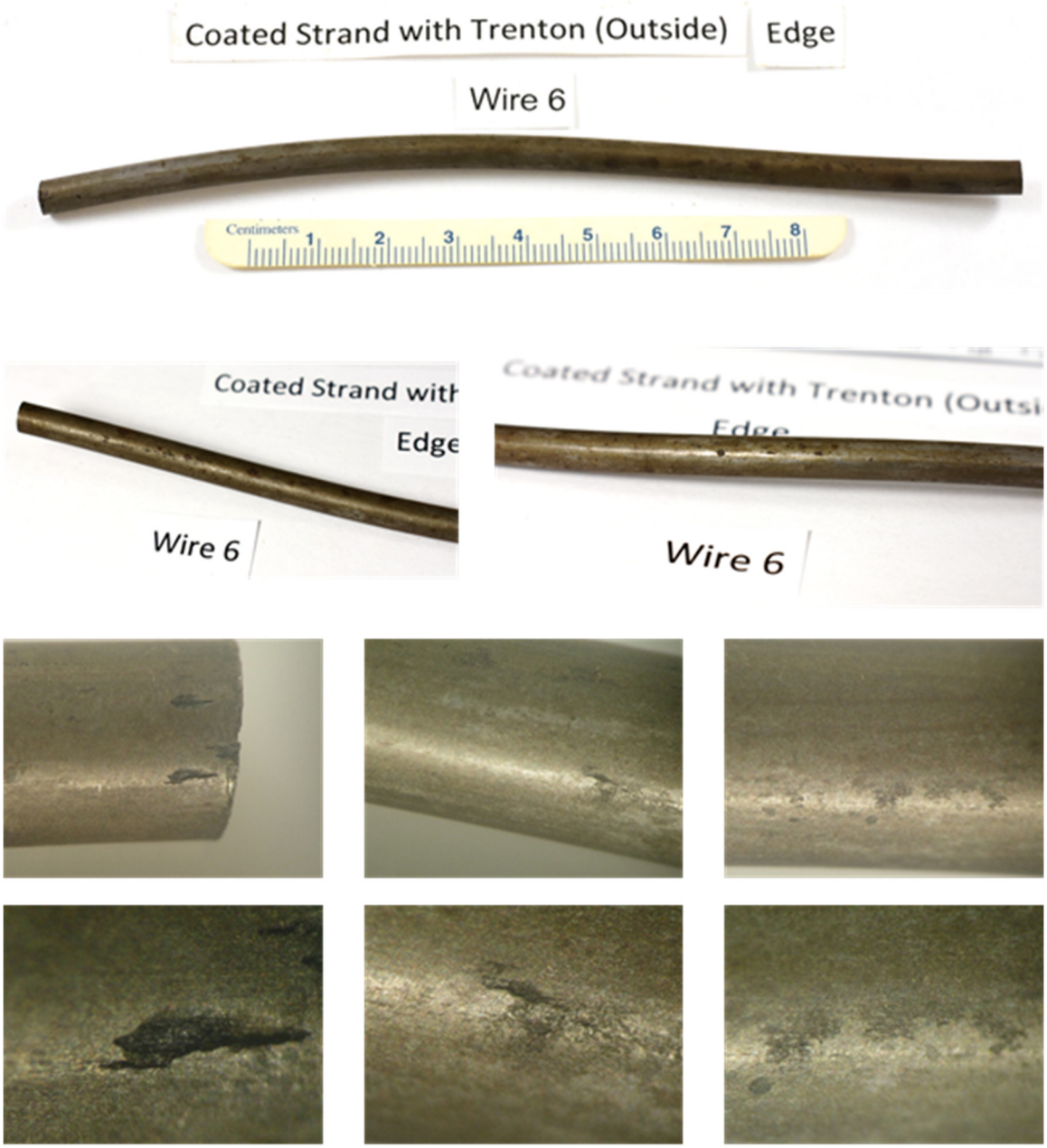


Figure 200. View of coated strand with Trenton (outside) edge , wire 6 after removing filler and cleaning (close-up 2)

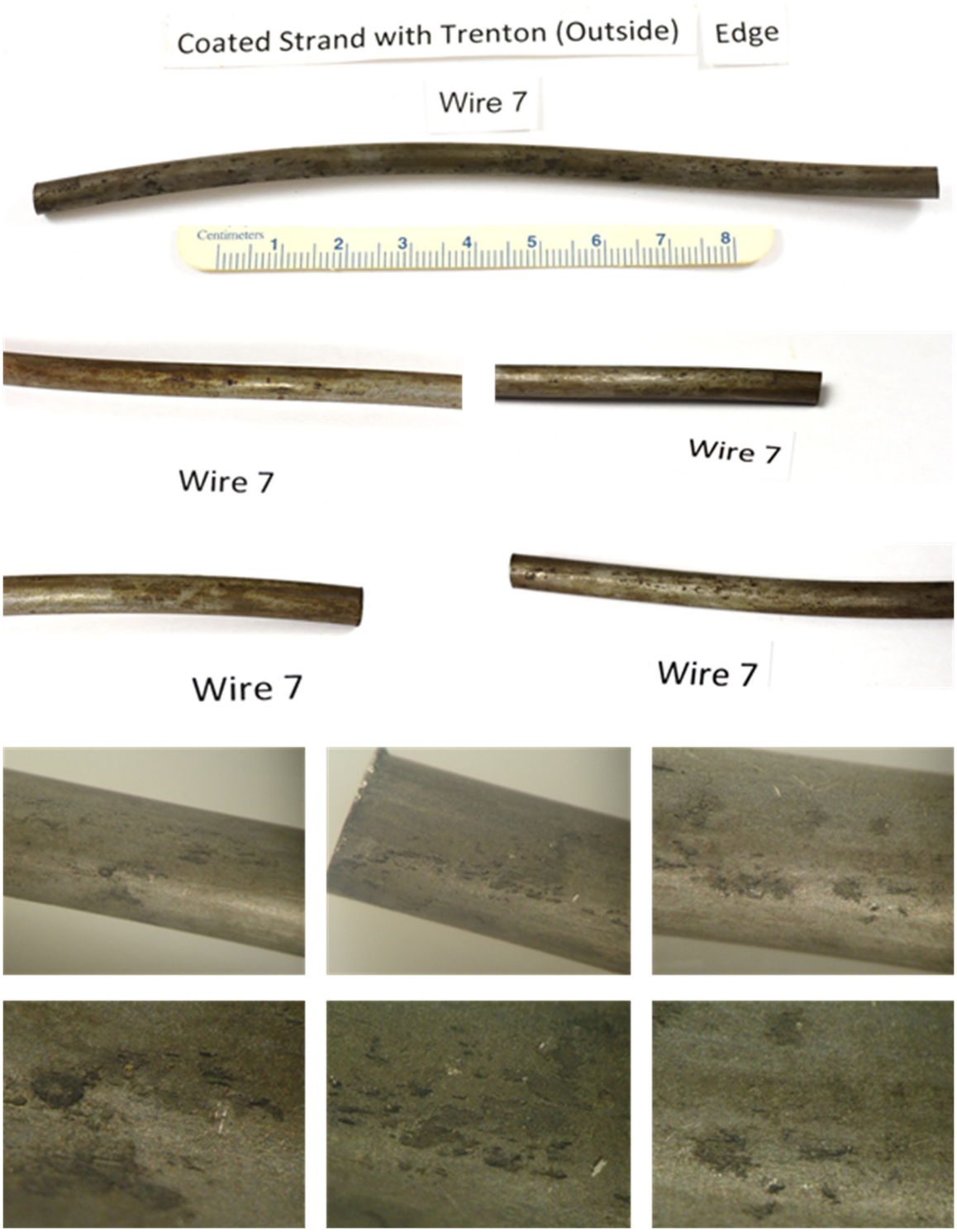


Figure 201. View of coated strand with Trenton (outside) edge , wire 7 after removing filler and cleaning (close-up 2)

Figure 202. View of coated strand with Trenton (outside) 2nd piece

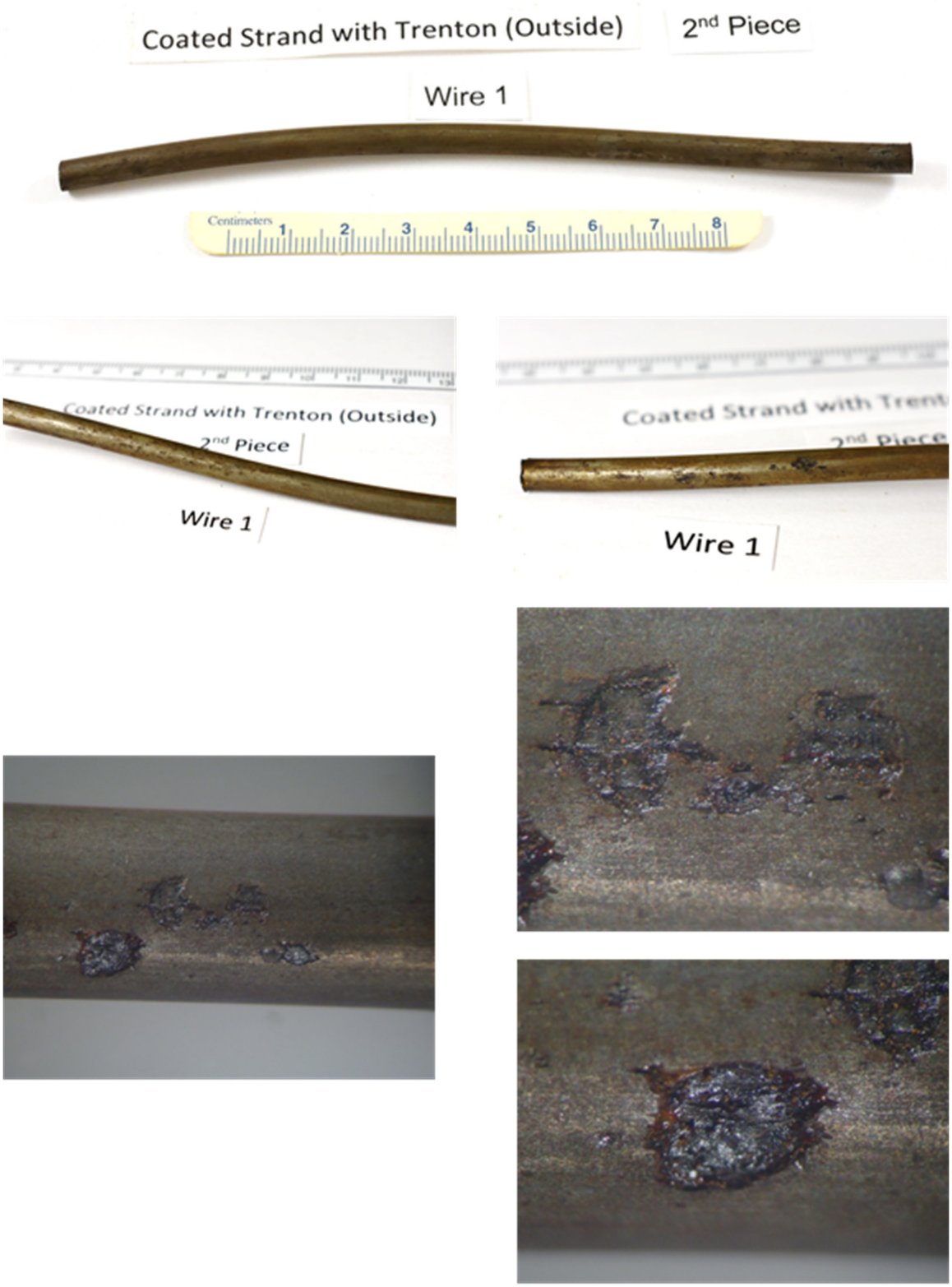


Figure 203. View of coated strand with Trenton (outside) 2nd piece, wire 1 after cleaning

Figure 204. View of coated strand with Trenton (outside) 2nd piece, wire 2 after cleaning

Figure 205. View of coated strand with Trenton (outside) 2nd piece, wire 3 after cleaning.

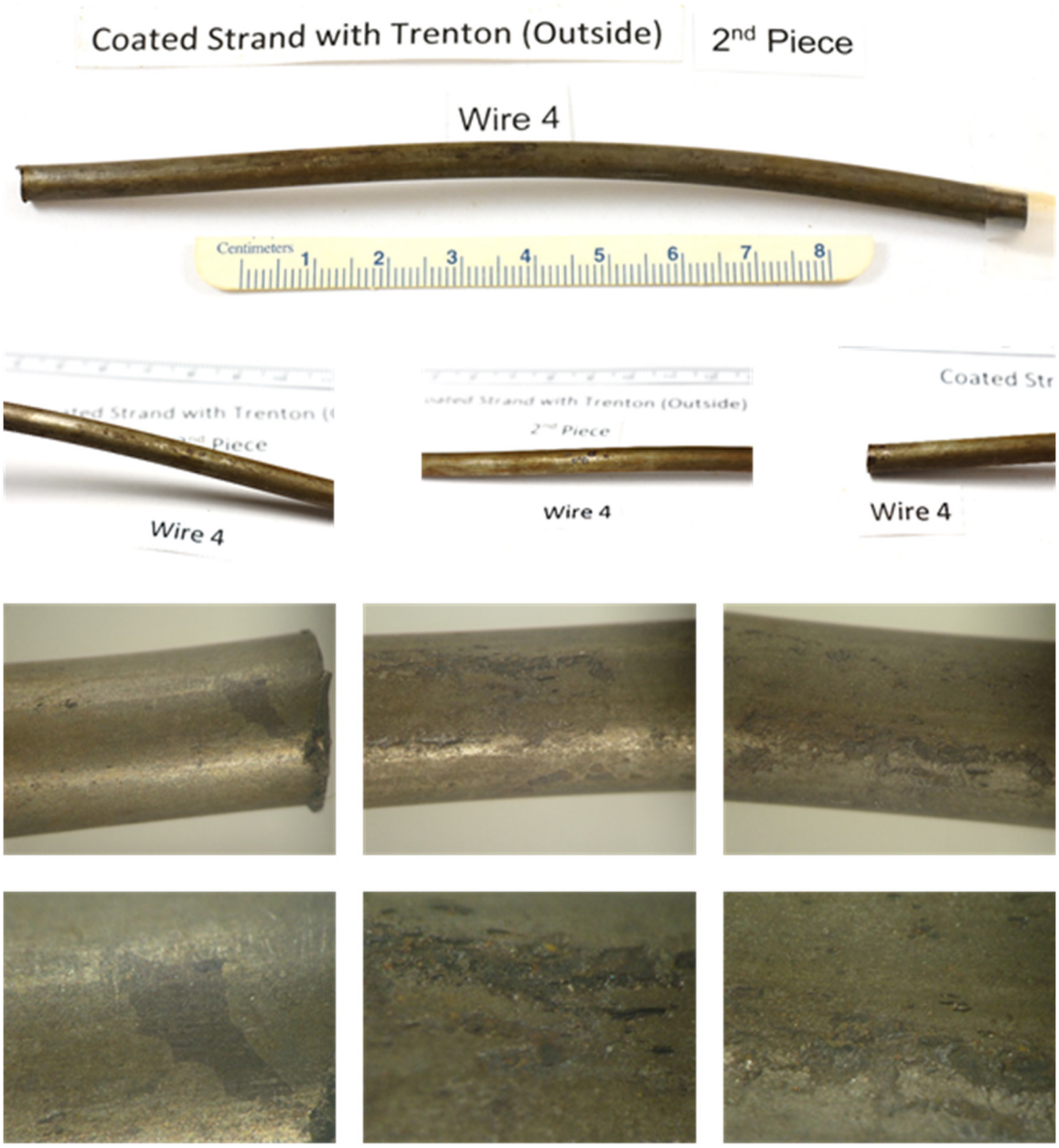


Figure 206. View of coated strand with Trenton (outside) 2nd piece, wire 4 after cleaning

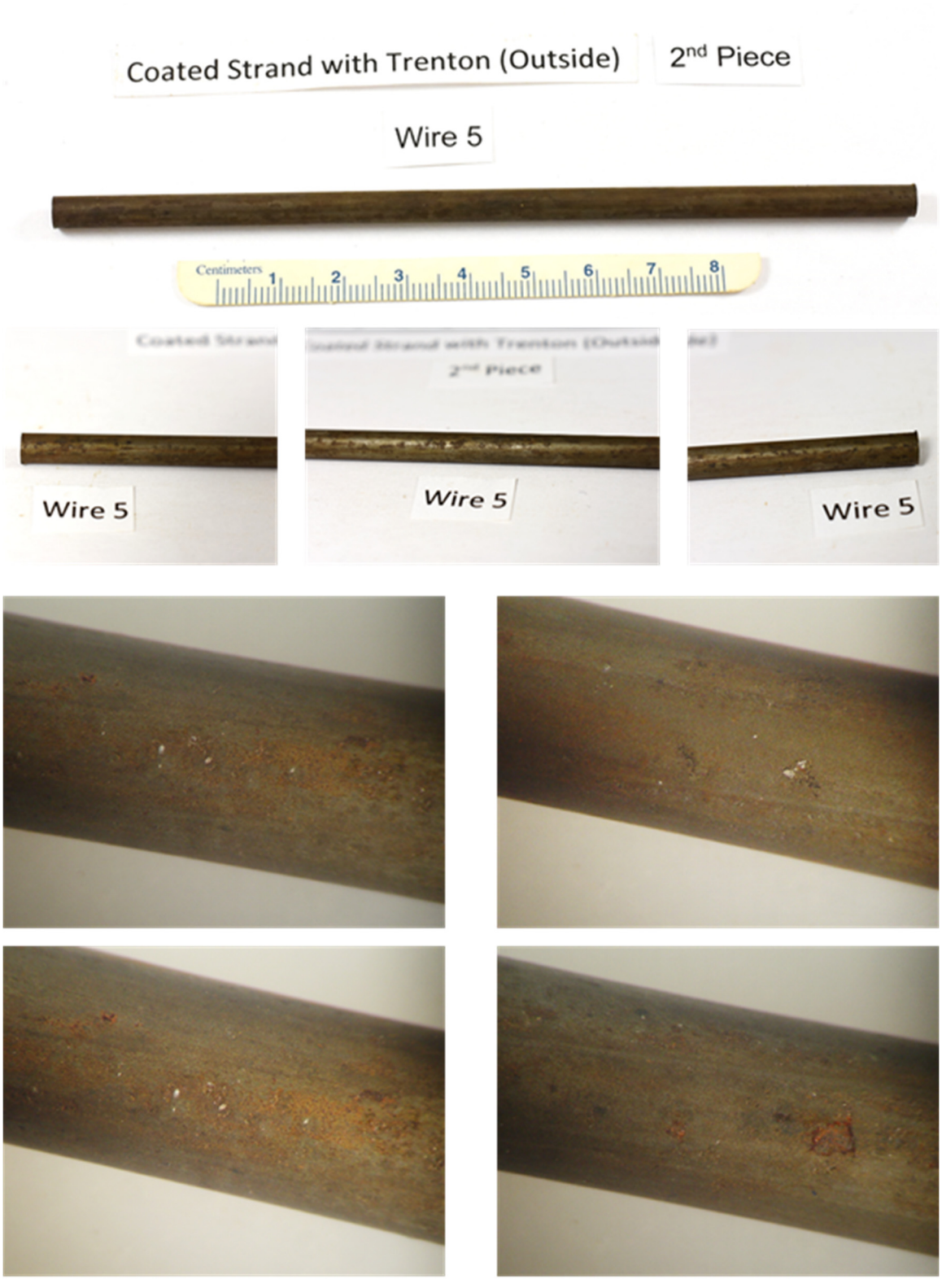


Figure 207. View of coated strand with Trenton (outside) 2nd piece , wire 5 after removing filler and cleaning (close-up).

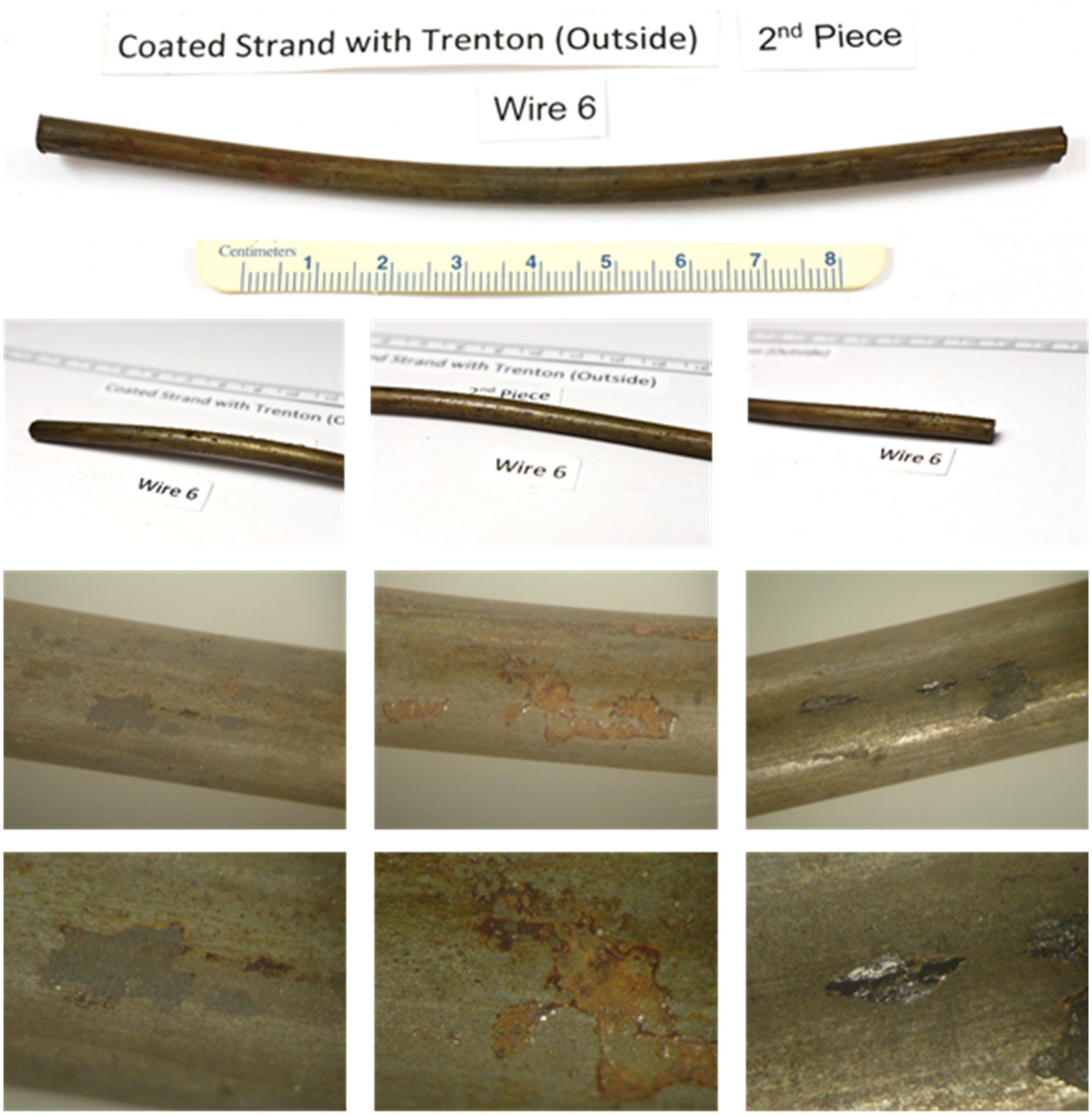
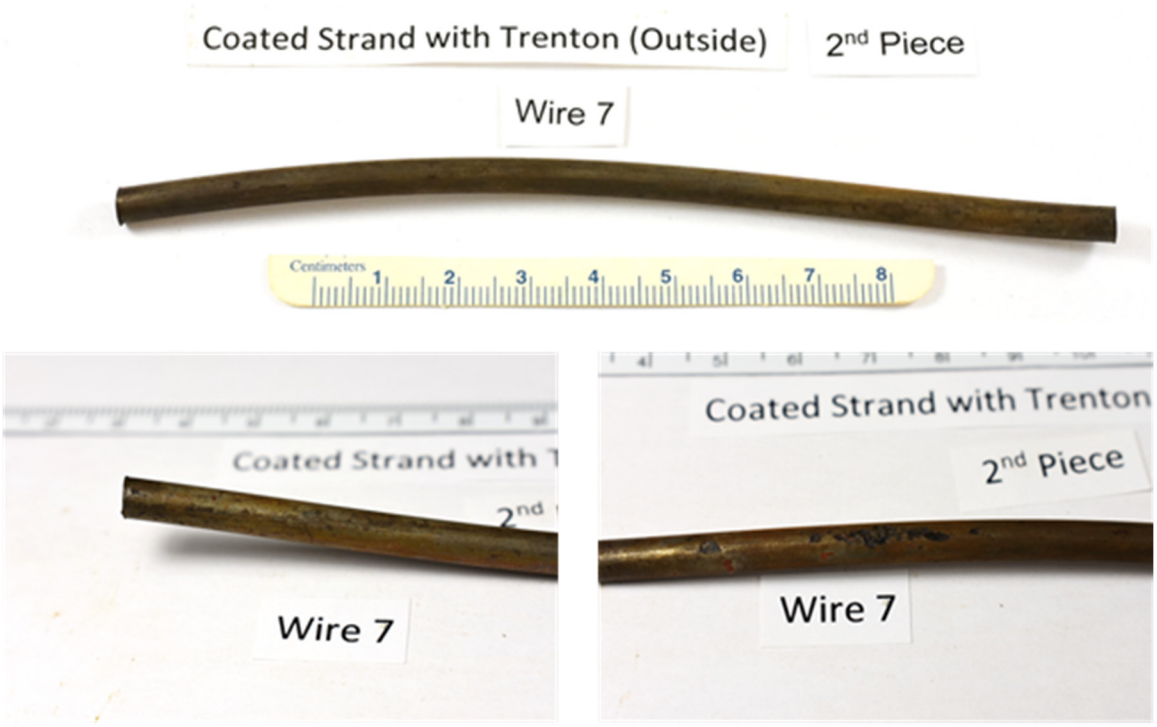


Figure 208. View of coated strand with Trenton (outside) 2nd piece , wire 6 after removing filler and cleaning (close-up).



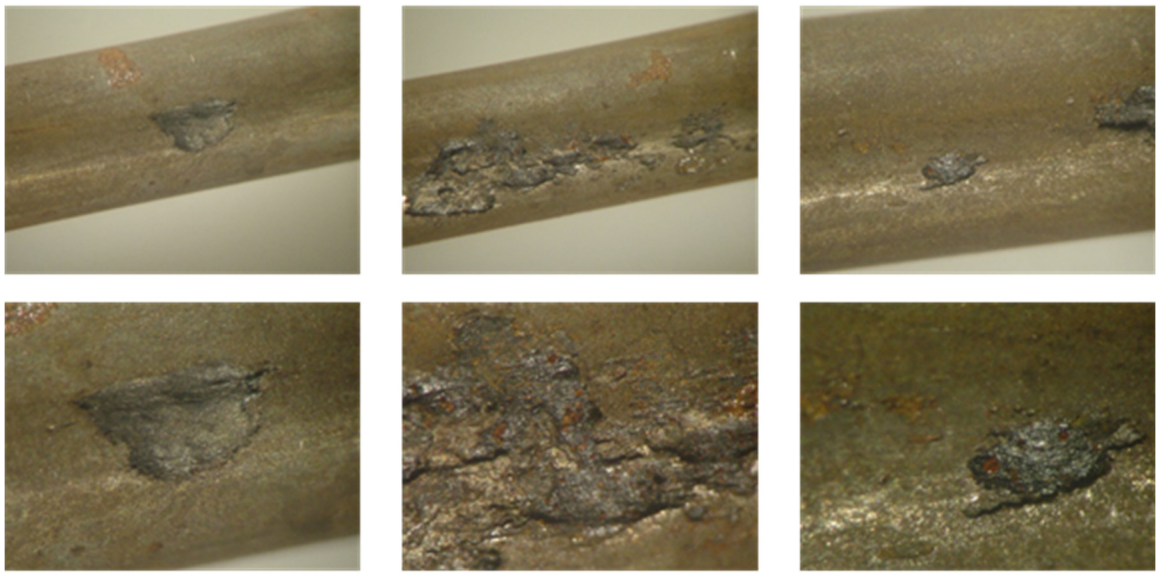


Figure 209. View of coated strand with Trenton (outside) 2nd piece , wire 7 after removing filler and cleaning (close-up 2).

Appendix B

Figure 210. Coated wires– exposed outside semi-sheltered area after cleaning

Figure 211. Single wire coated with Trenton exposed outside semi-sheltered area.

Figure 212. Single wire coated with Nontribos exposed outside semi-sheltered area

Figure 213. Single wire coated with Sonneborn exposed outside semi-sheltered area

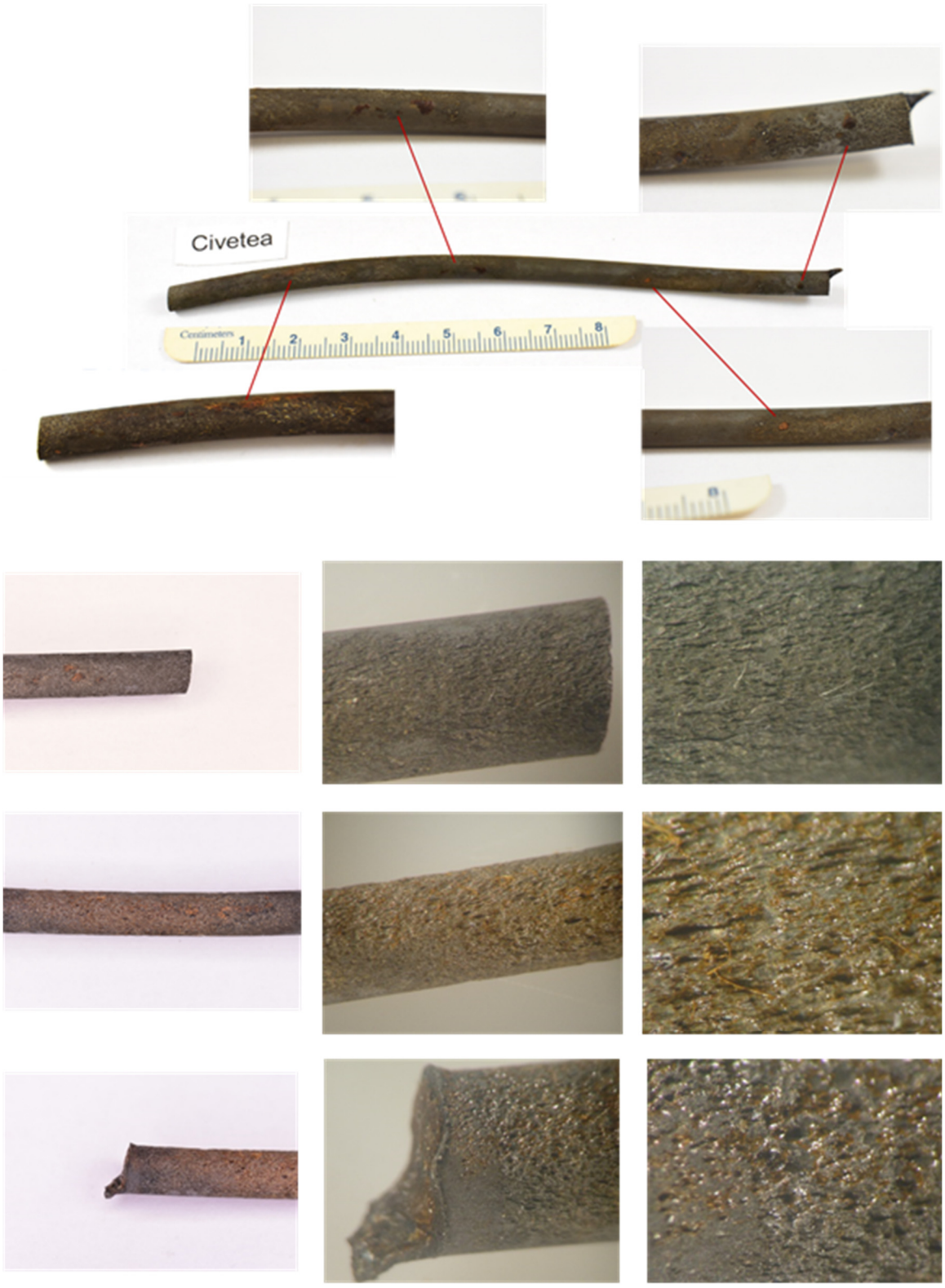


Figure 214. Single wire coated with Civetea exposed outside semi-sheltered area

Figure 215. Single wire coated with Trenton exposed outdoors inside partially closed container

Figure 216. Single wire coated with Nontribos exposed outdoors inside partially closed container

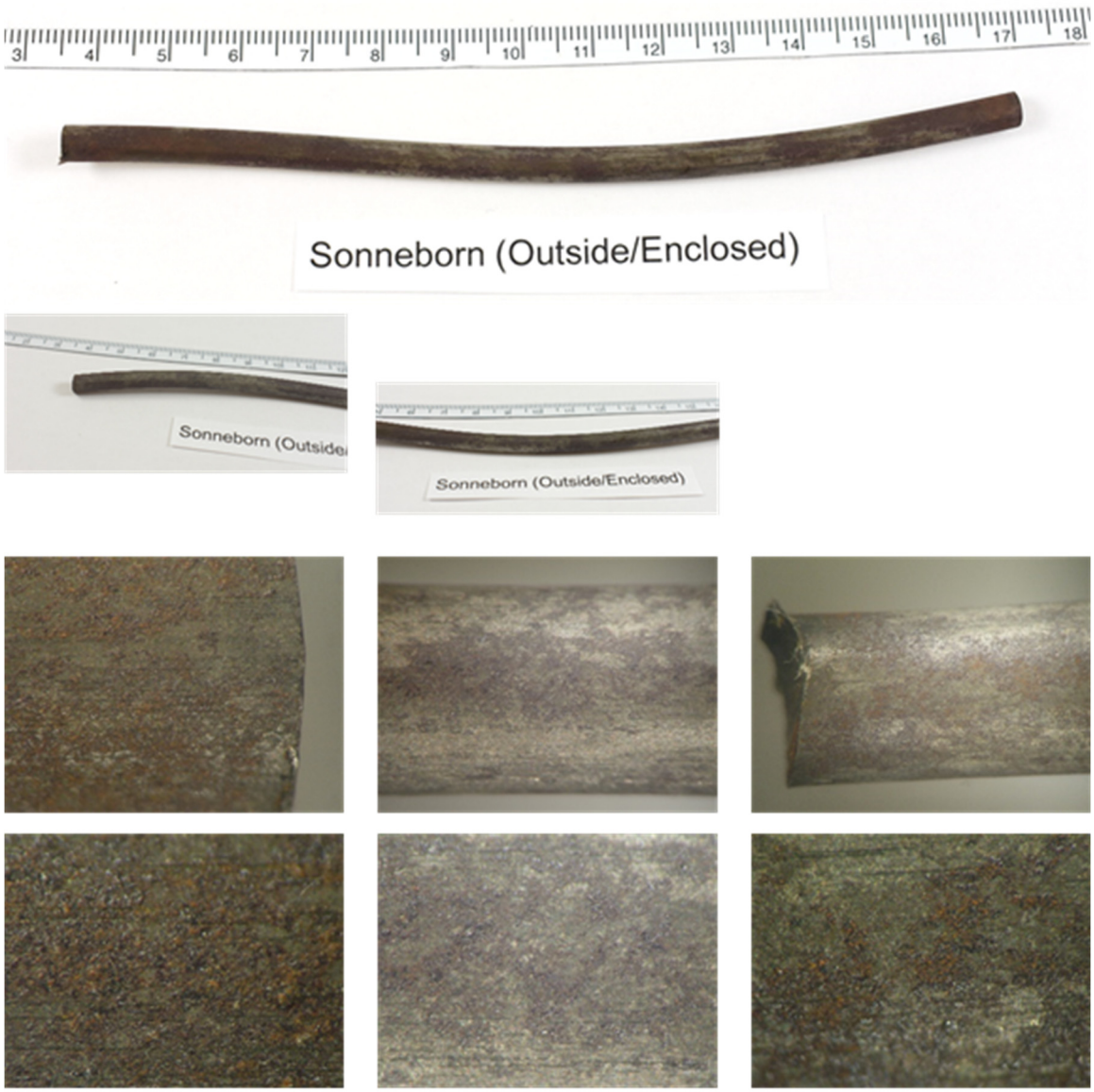


Figure 217. Single wire coated with Sonneborn exposed outdoors inside partially closed container (close-up 2)

Figure 218. Single wire coated with Civetea exposed outdoors inside partially closed container.

Figure 219. Single wire coated with Sanchem exposed outdoors inside partially closed container.

Figure 220. Single wire coated with Trenton at 20°C at high humidity.

Figure 221. Single wire coated with Nontribos at 20°C at high humidity

Figure 222. Single wire coated with Sonneborn at 20°C at high humidity

Figure 223. Single wire coated with Civetea at 20°C at high humidity

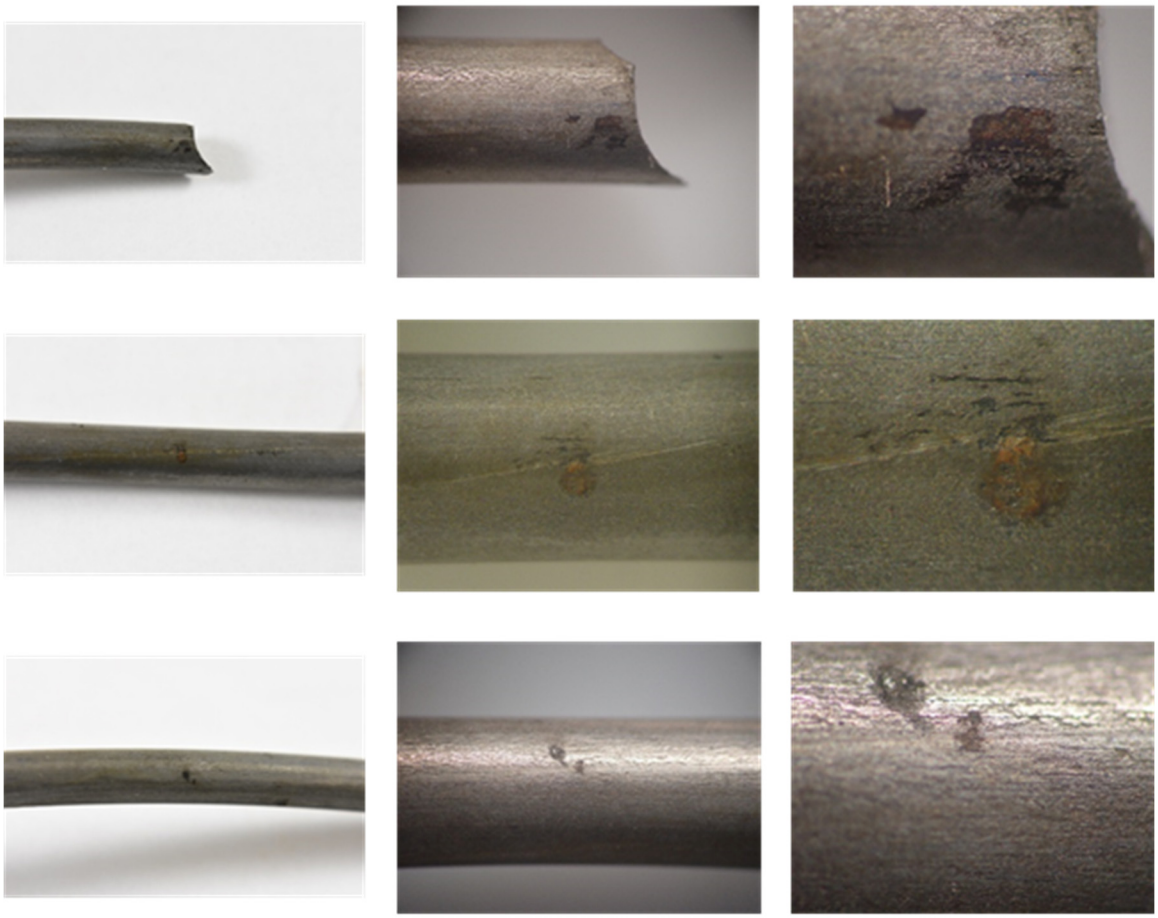


Figure 224. Single wire coated with Sanchem at 20°C at high humidity

Figure 225. Single wire blank at 20°C at high humidity

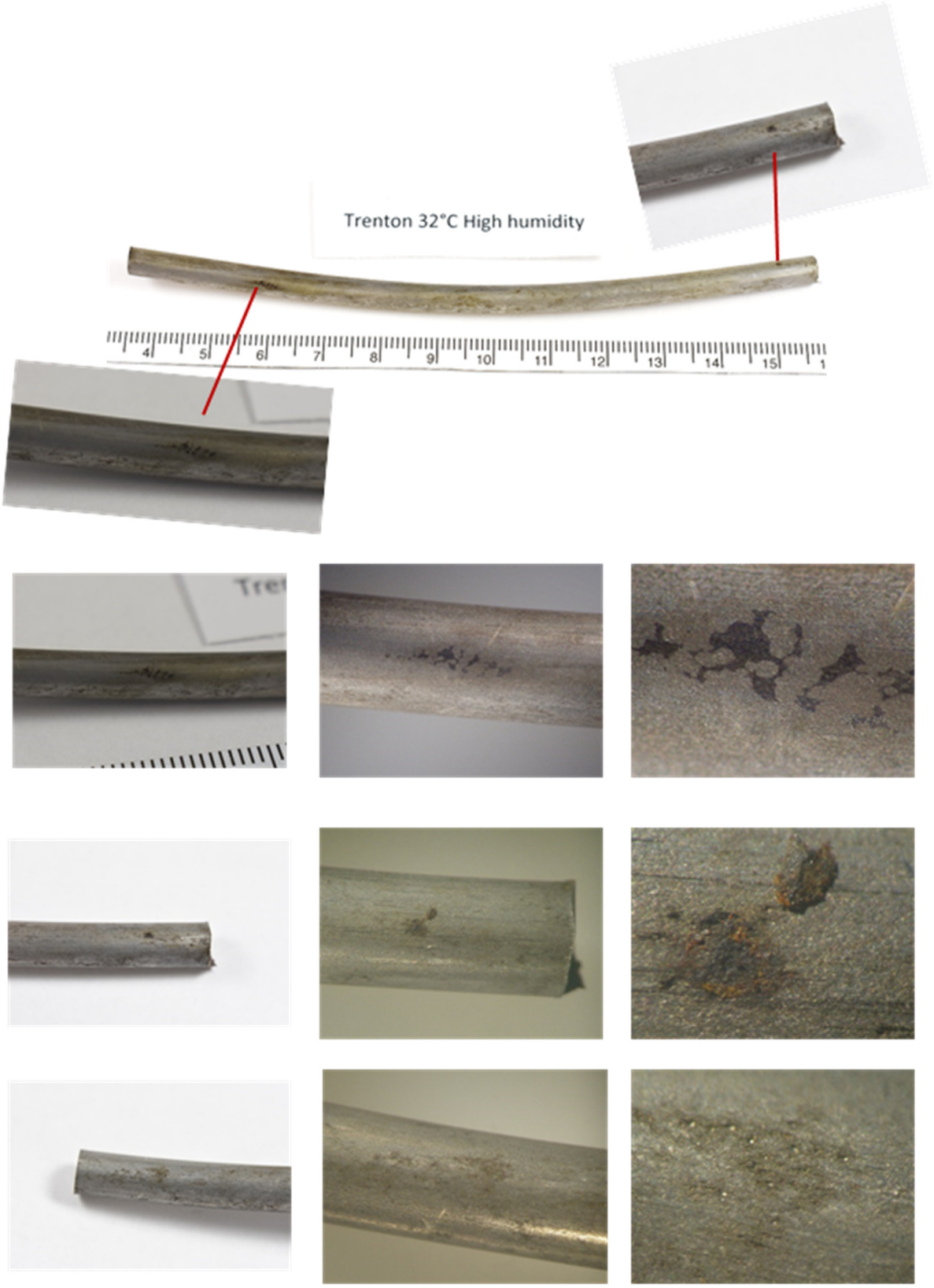


Figure 226. Single wire coated with Trenton at 32°C at high humidity (close-up 1)

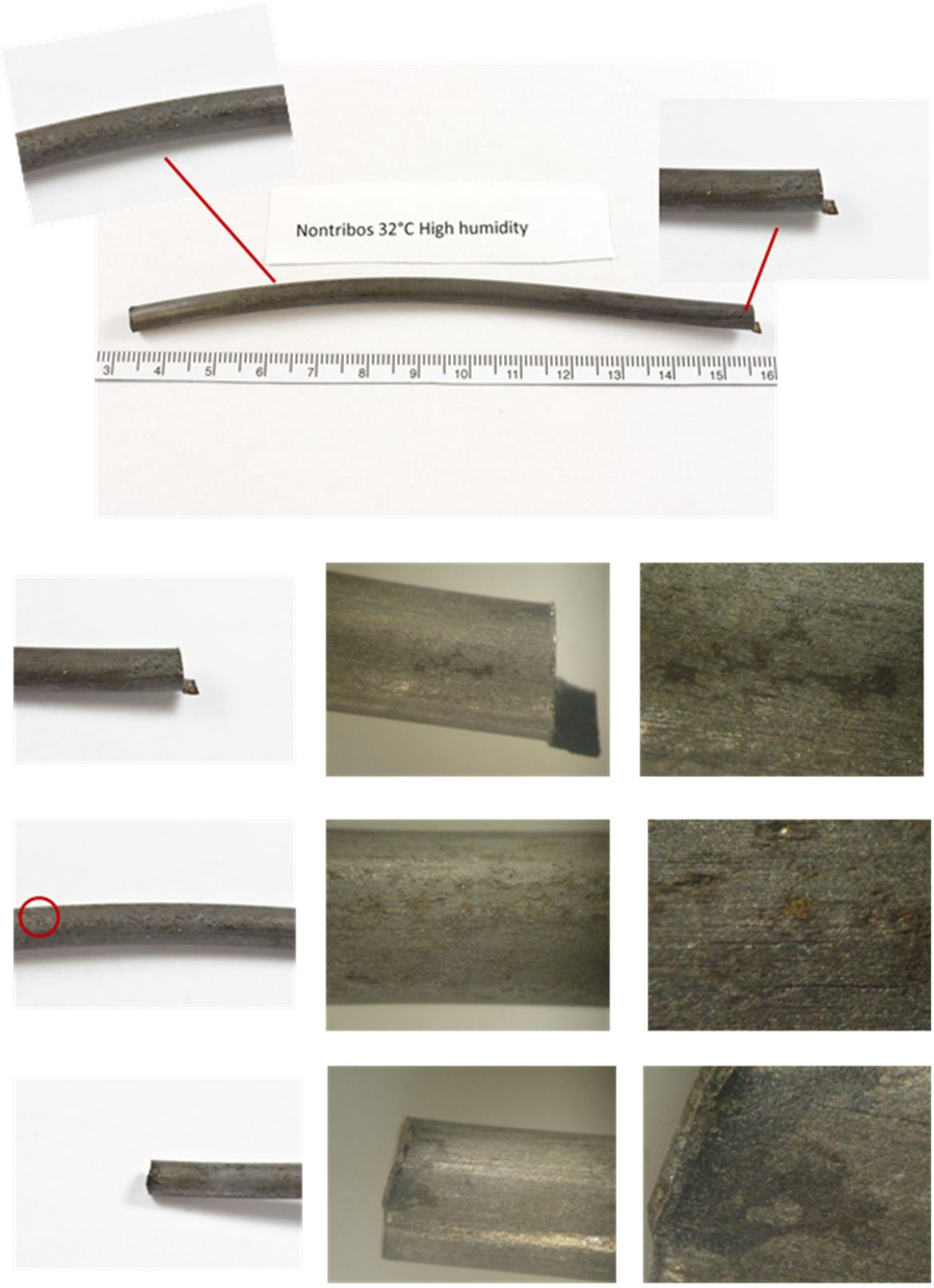


Figure 227. Single wire coated with Nontribos at 32°C at high humidity

Figure 228. Single wire coated with Sonneborn at 32°C at high humidity

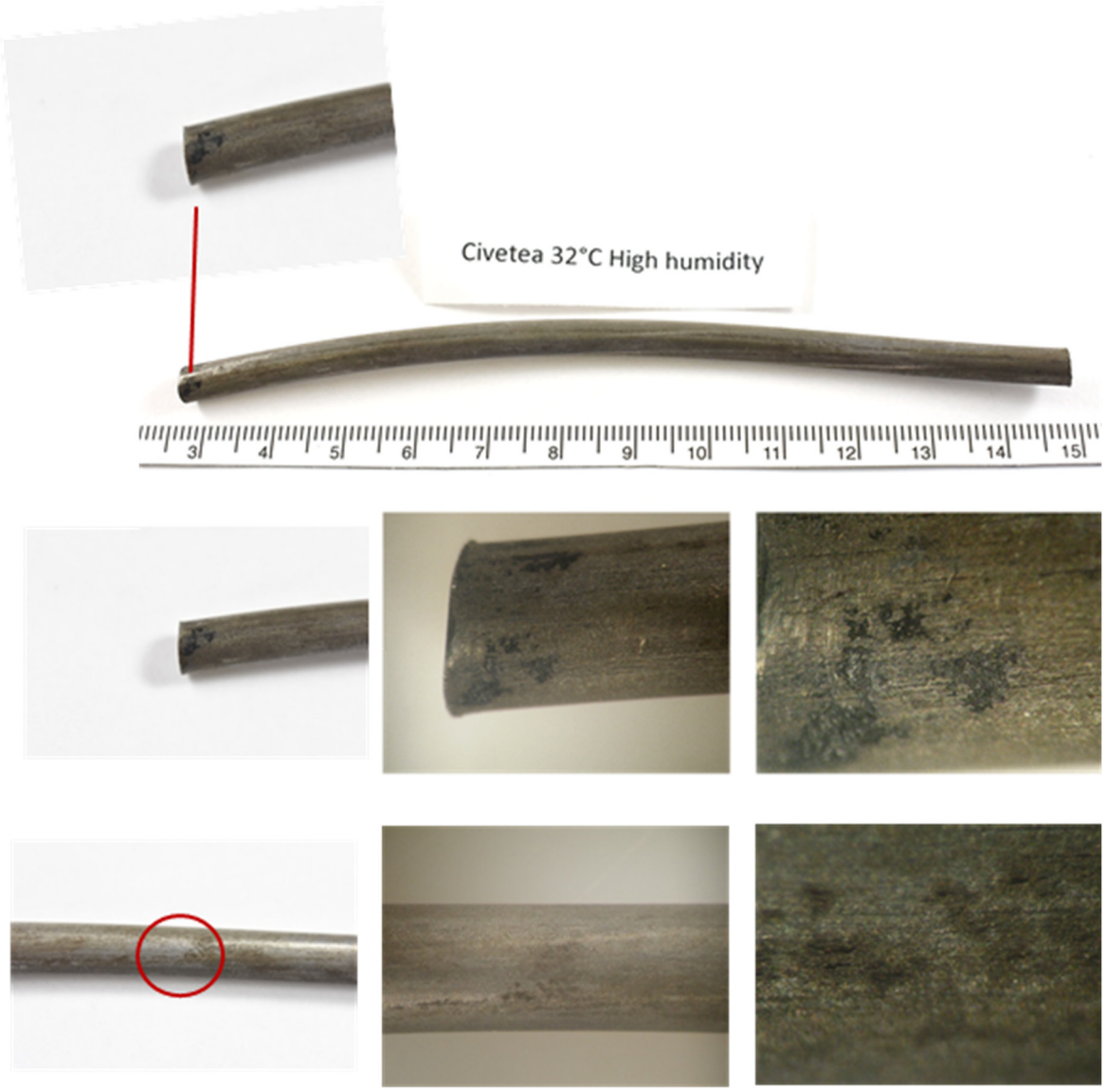


Figure 229. Single wire coated with Civetea at 32°C at high humidity.

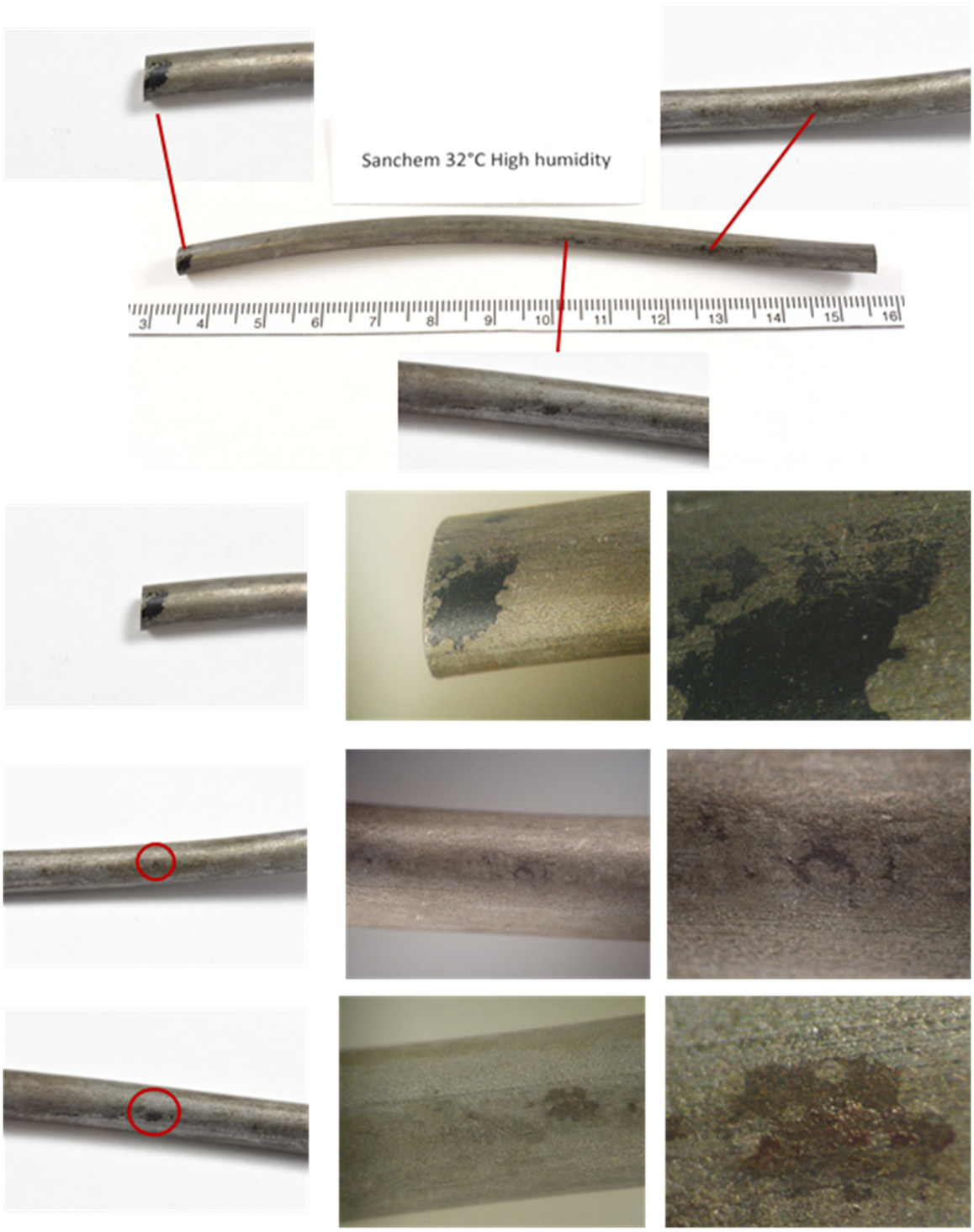


Figure 230. Single wire coated with Sanchem at 32°C at high humidity

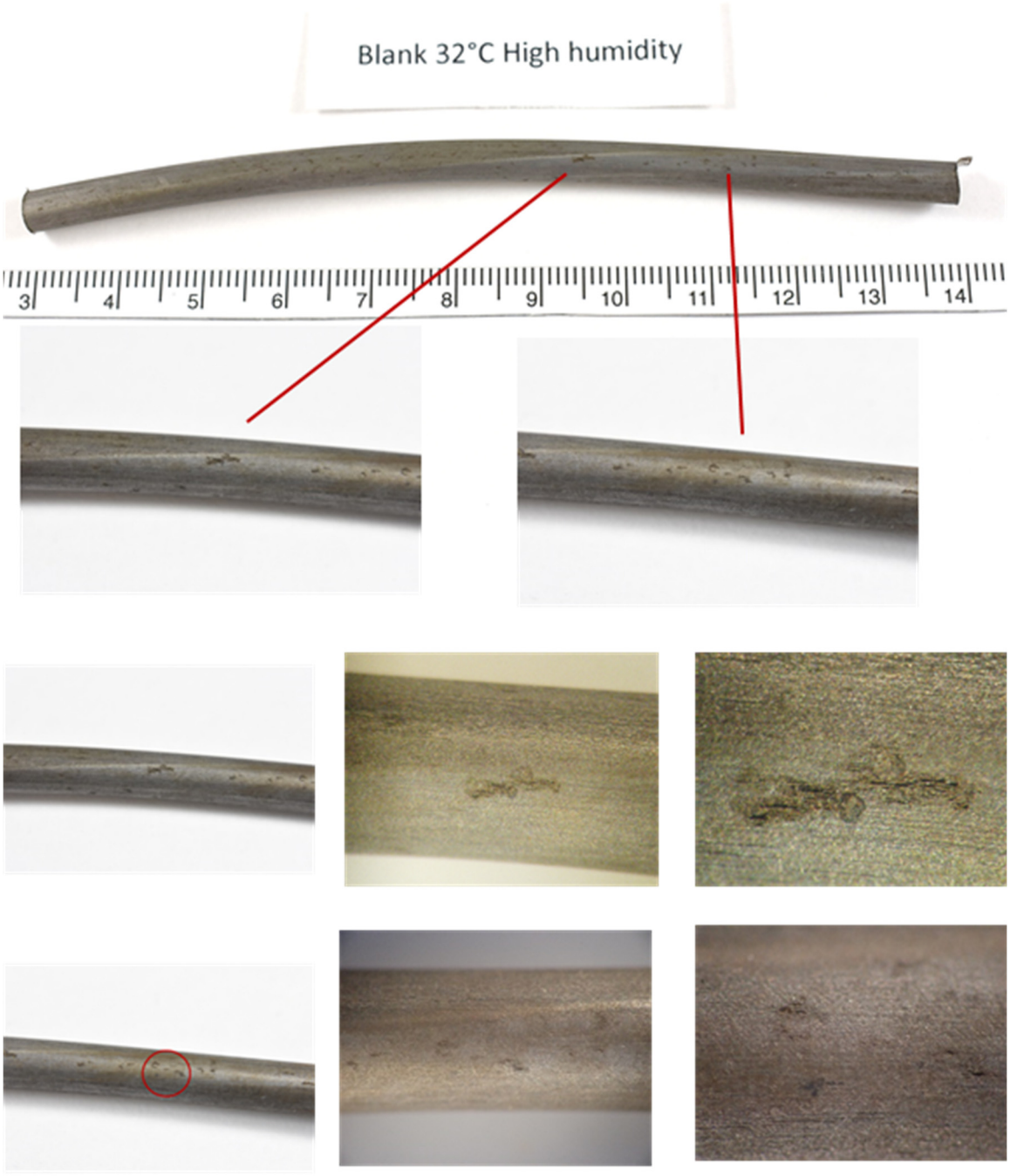


Figure 231. Single wire blank at 32°C at high humidity

Appendix C

Figure 232. Coated with filler and sprayed with 1.5 milliliter of solution containing fungus AF

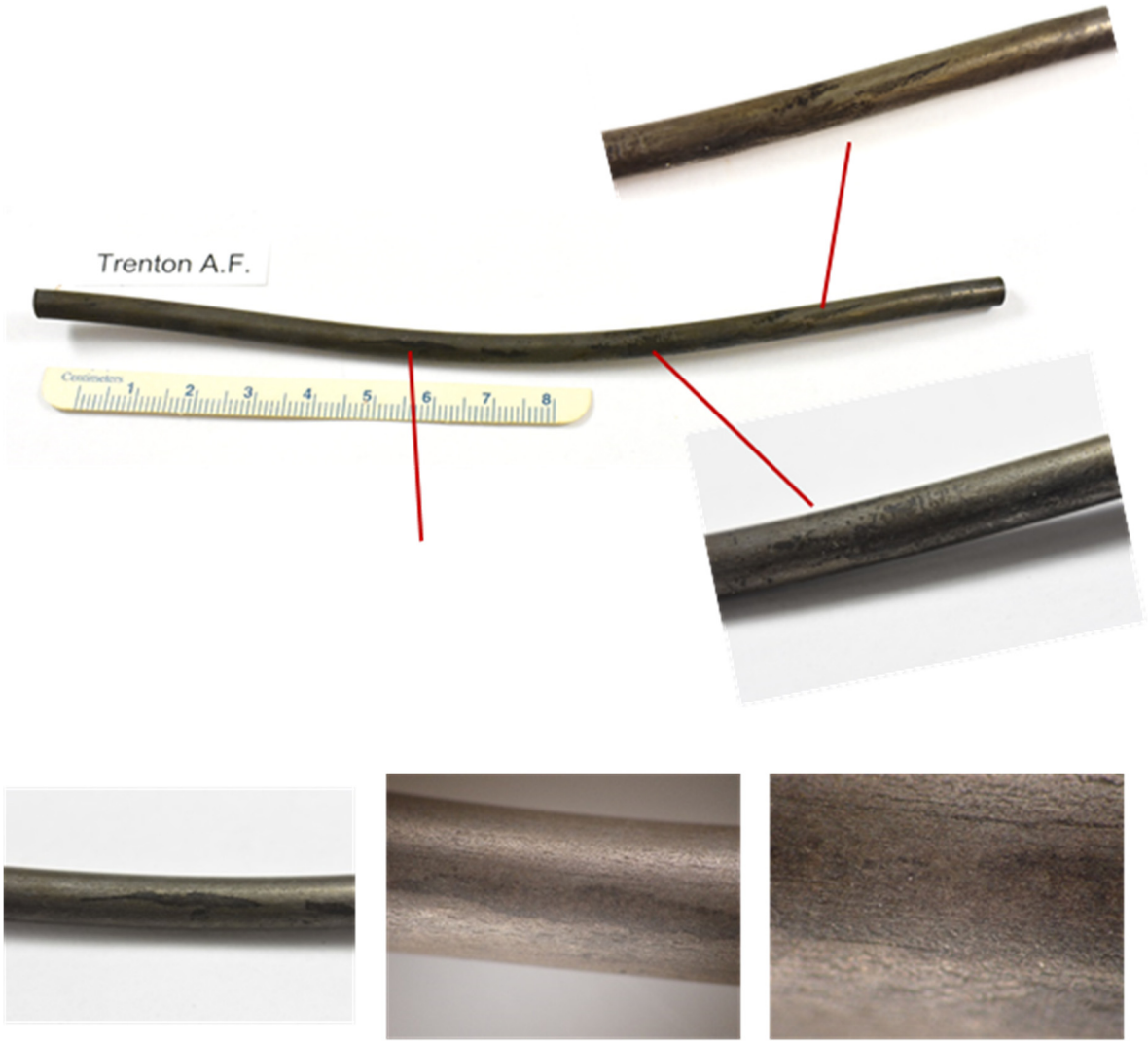


Figure 233. Coated with filler Trenton and sprayed with 1.5 milliliter of solution containing fungus AF

Figure 234. Coated with filler Trenton and sprayed with 1.5 milliliter of solution containing fungus AF

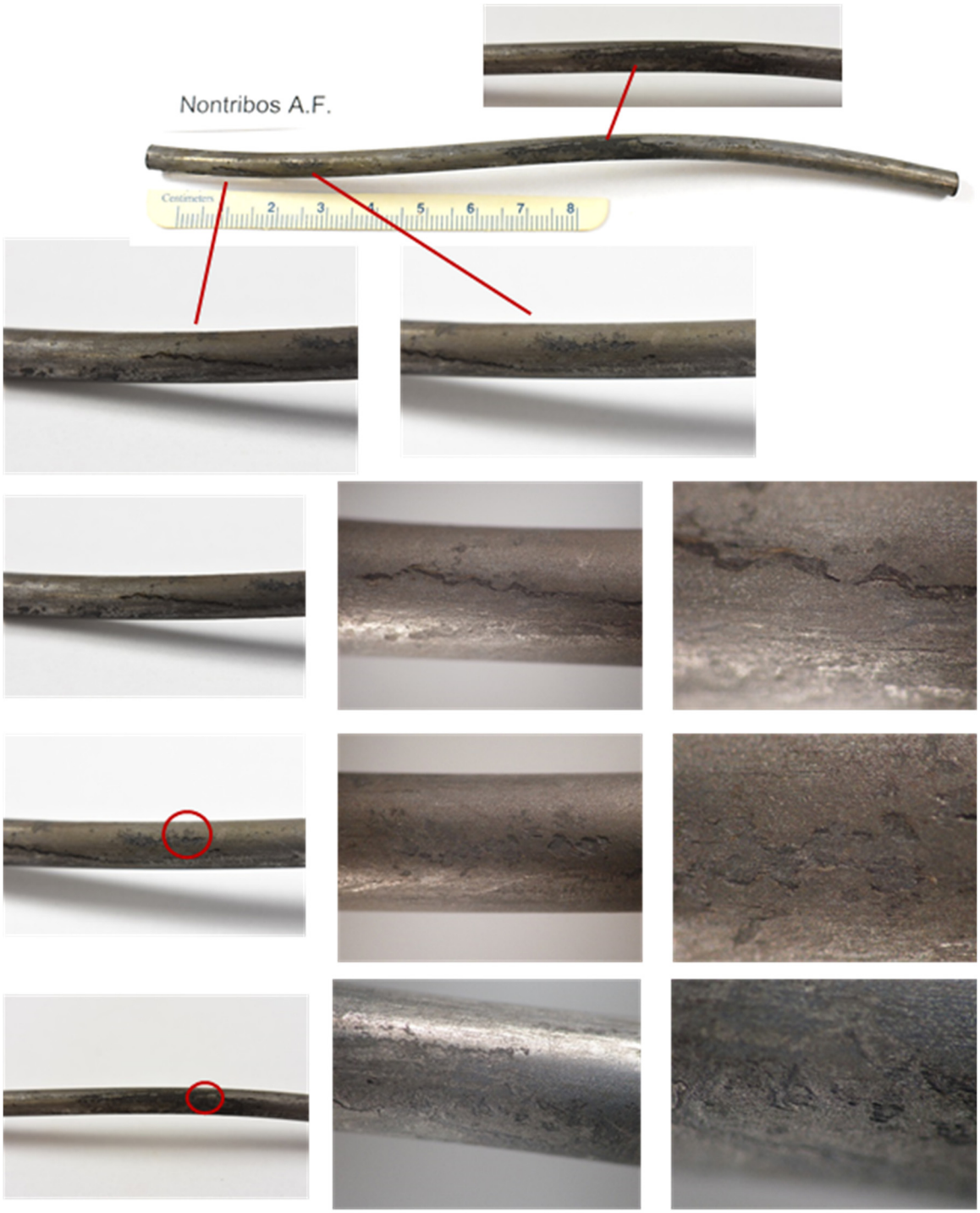


Figure 235. Coated with filler Nontribos and sprayed with 1.5 milliliter of solution containing fungus AF

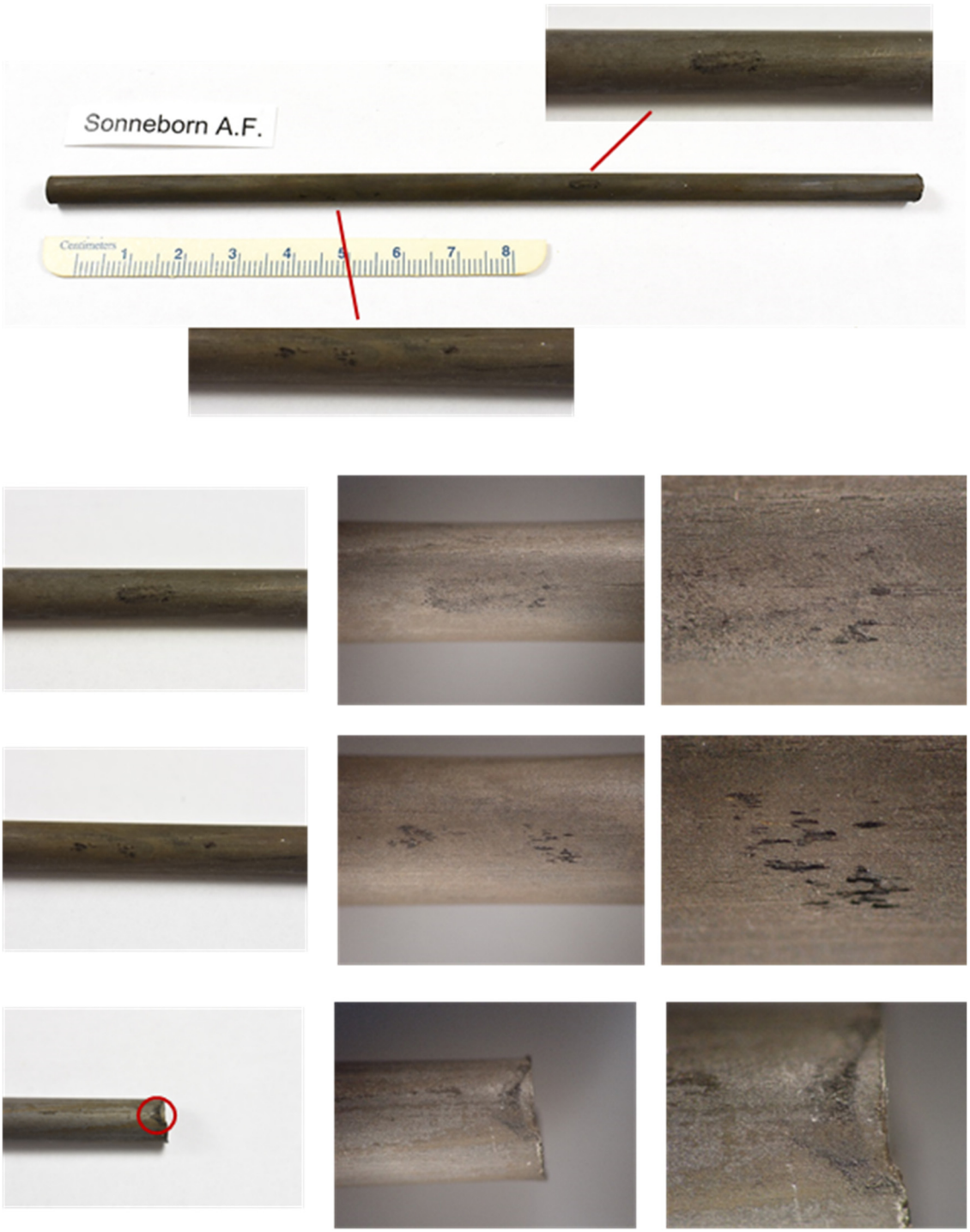


Figure 236. Coated with filler Sonneborn and sprayed with 1.5 milliliter of solution containing

fungus AF

Figure 237. Coated with filler Sanchem and sprayed with 1.5 milliliter of solution containing fungus AF (close-up 3).

Figure 238. Coated with filler and sprayed with 1.5 milliliter of solution containing fungus FO

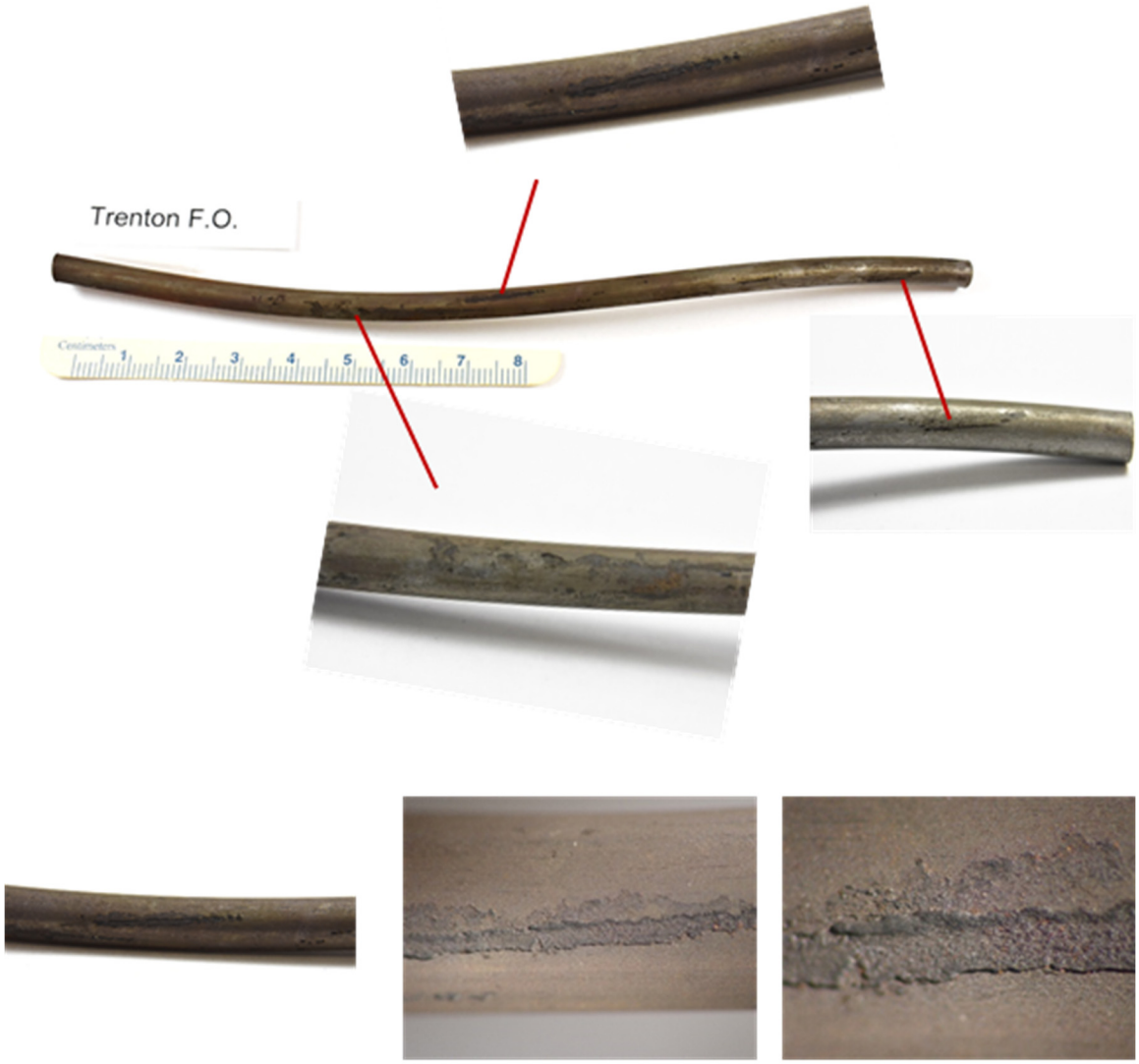


Figure 239. Coated with filler Trenton and sprayed with 1.5 milliliter of solution containing fungus FO (close-up 1)

Figure 240. Coated with filler Trenton and sprayed with 1.5 milliliter of solution containing fungus FO (close-up 2)

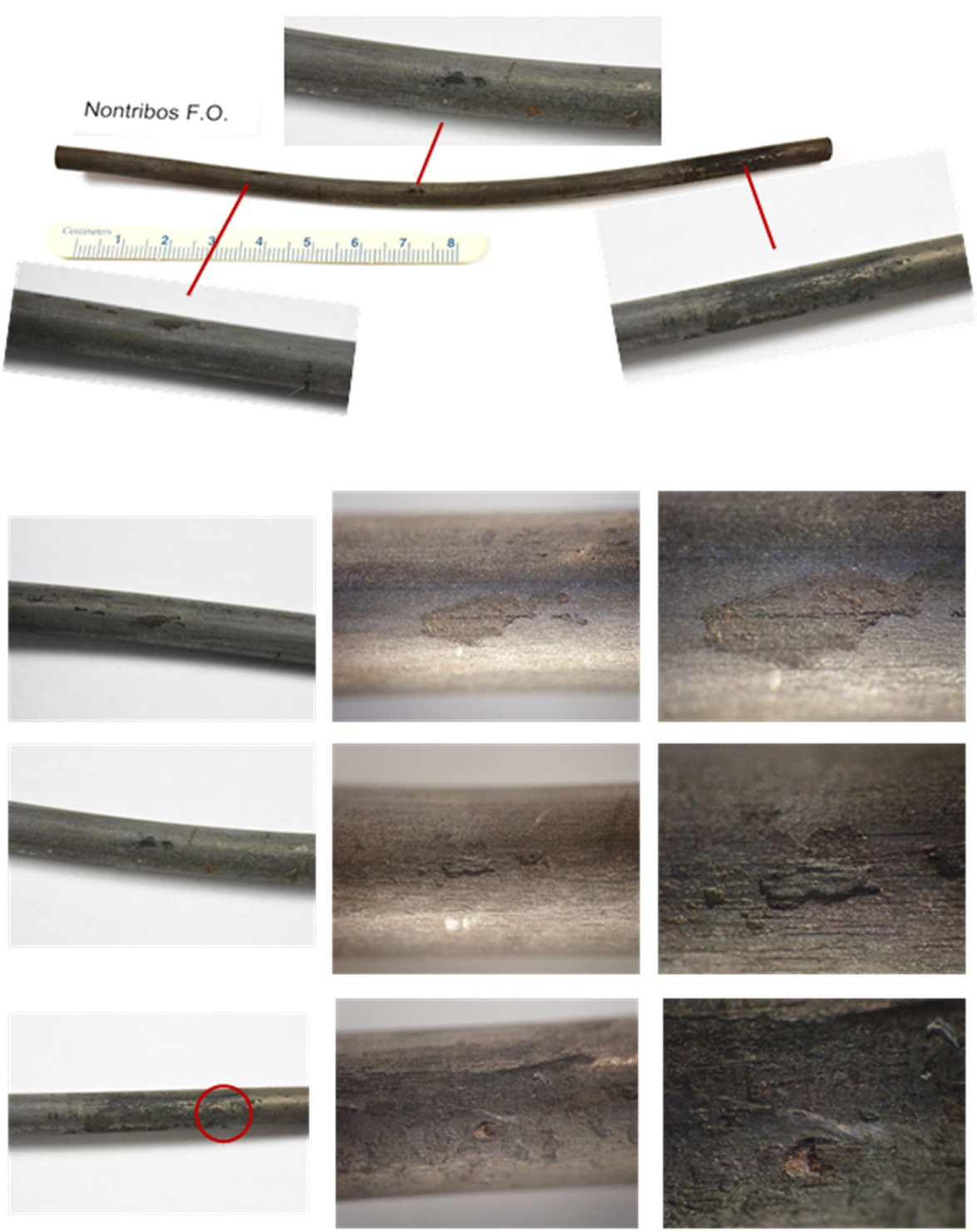


Figure 241. Coated with filler Nontribos and sprayed with 1.5 milliliter of solution containing fungus FO (close-up 3)

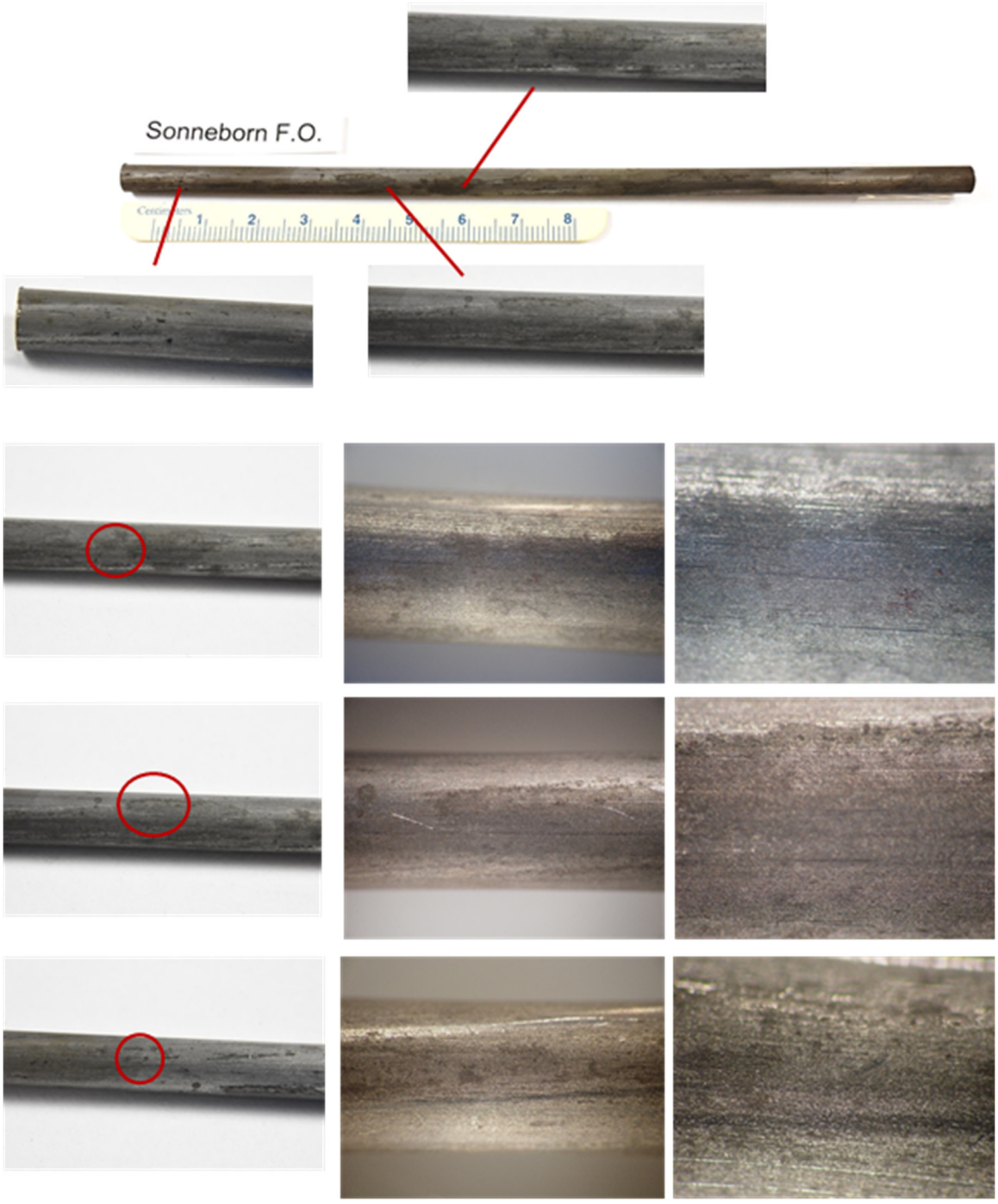


Figure 242. Coated with filler Sonneborn and sprayed with 1.5 milliliter of solution containing fungus FO (close-up)

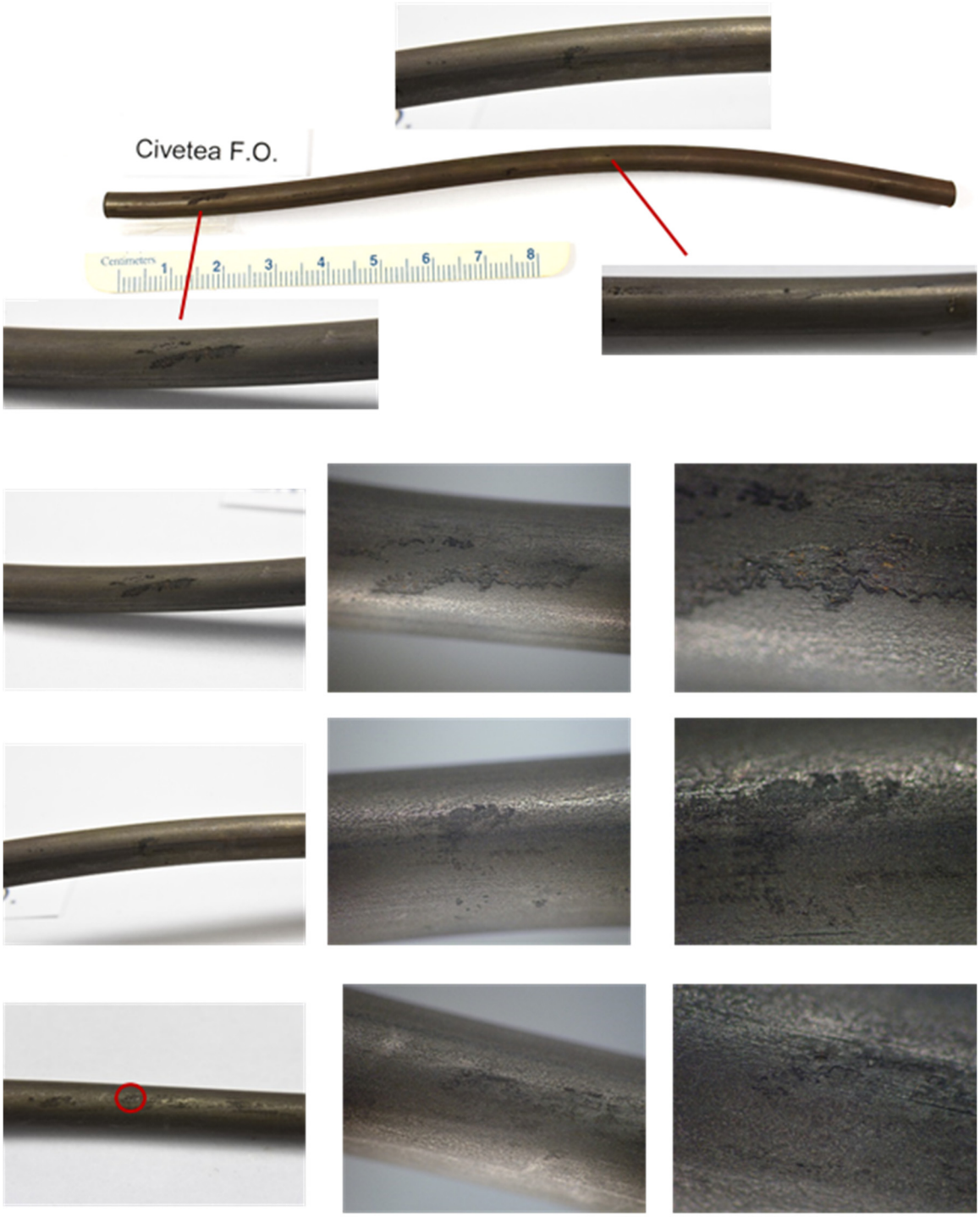


Figure 243. Coated with filler Civetea and sprayed with 1.5 milliliter of solution containing fungus FO (close-up)

Figure 244. Coated with filler Sanchem and sprayed with 1.5 milliliter of solution containing fungus FO (close-up).

Figure 245. Coated with filler Sanchem and sprayed with 1.5 milliliter of solution containing fungus FO (close-up 2).

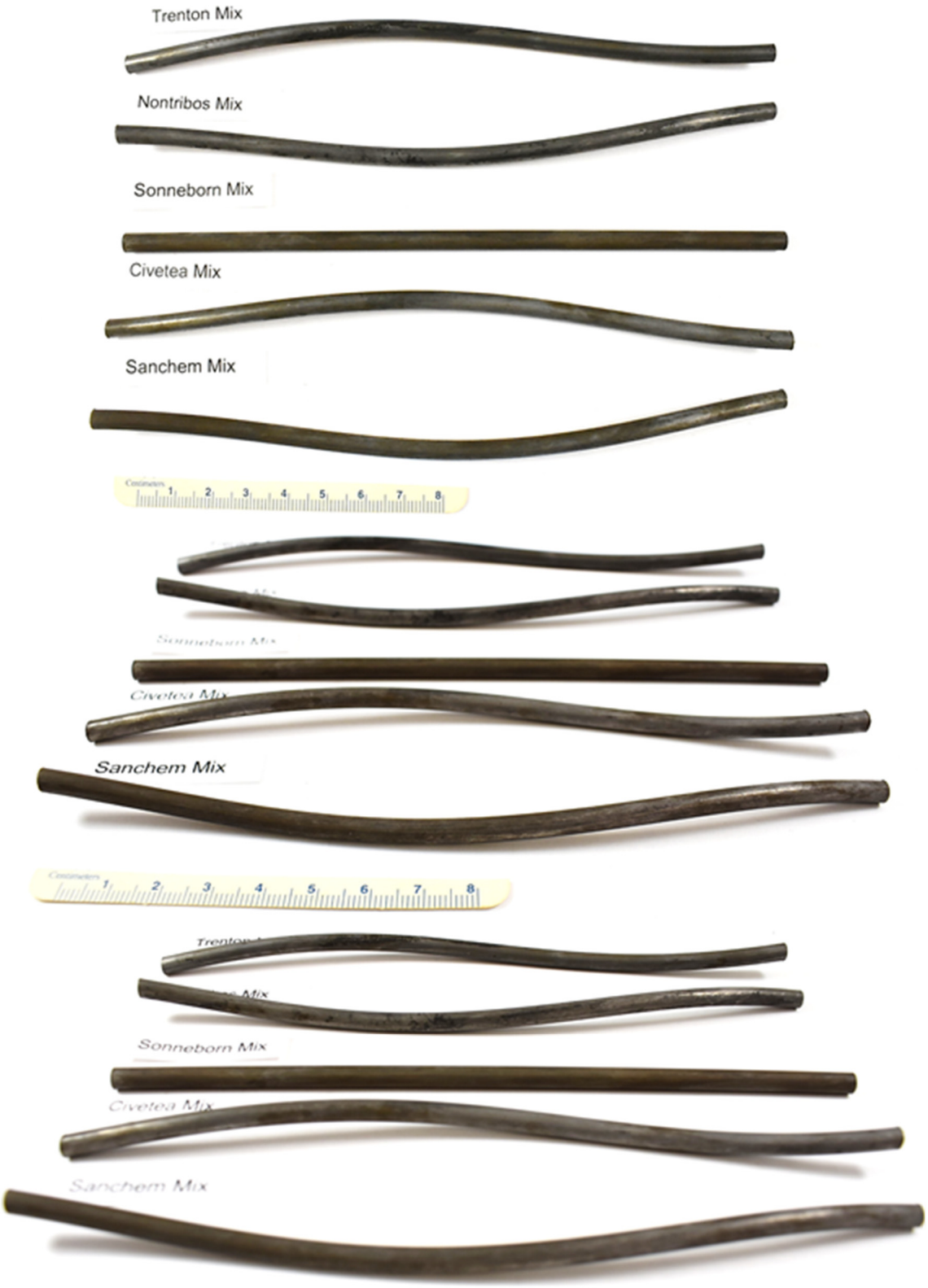


Figure 246. Coated with filler and sprayed with 1.5 milliliter of solution containing fungi mix.

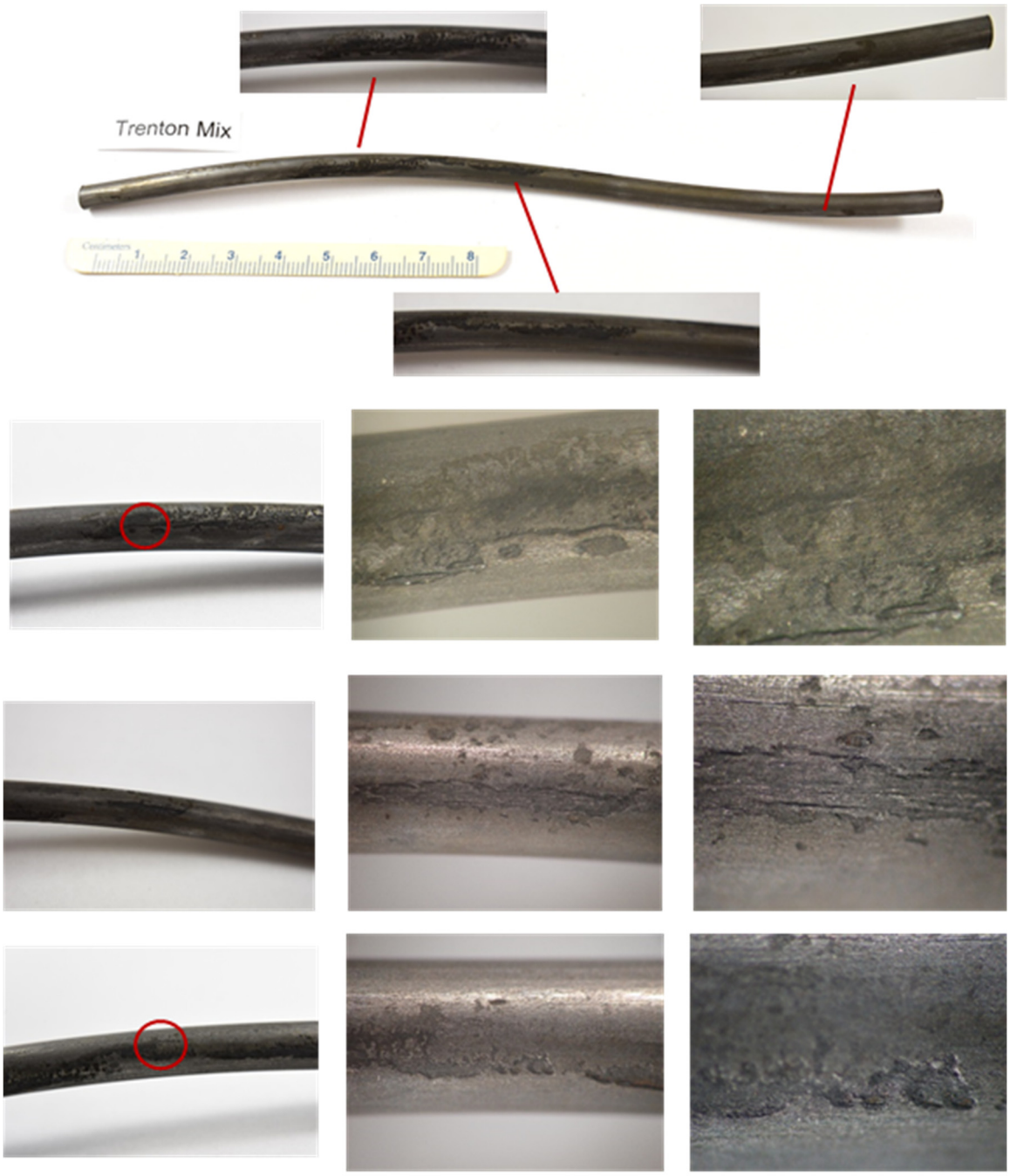


Figure 247. Coated with filler Trenton and sprayed with 1.5 milliliter of solution containing fungi mix (close-up).

Figure 248. Coated with filler Nontribos and sprayed with 1.5 milliliter of solution containing fungi mix (close-up).

Figure 249. Coated with filler Sonneborn and sprayed with 1.5 milliliter of solution containing fungi mix (close-up).

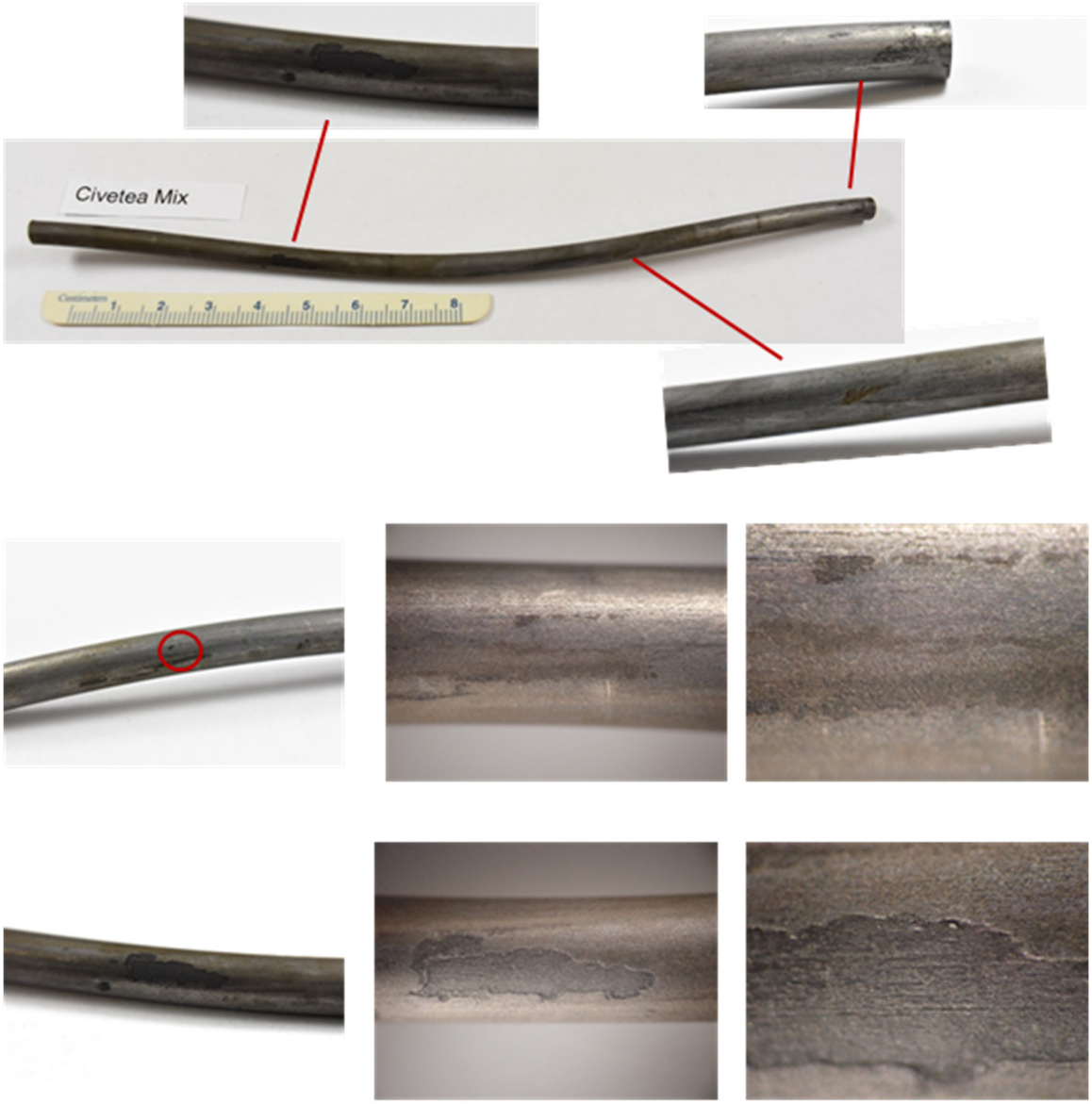


Figure 250. Coated with filler Civetea and sprayed with 1.5 milliliter of solution containing fungi mix (close-up 1).

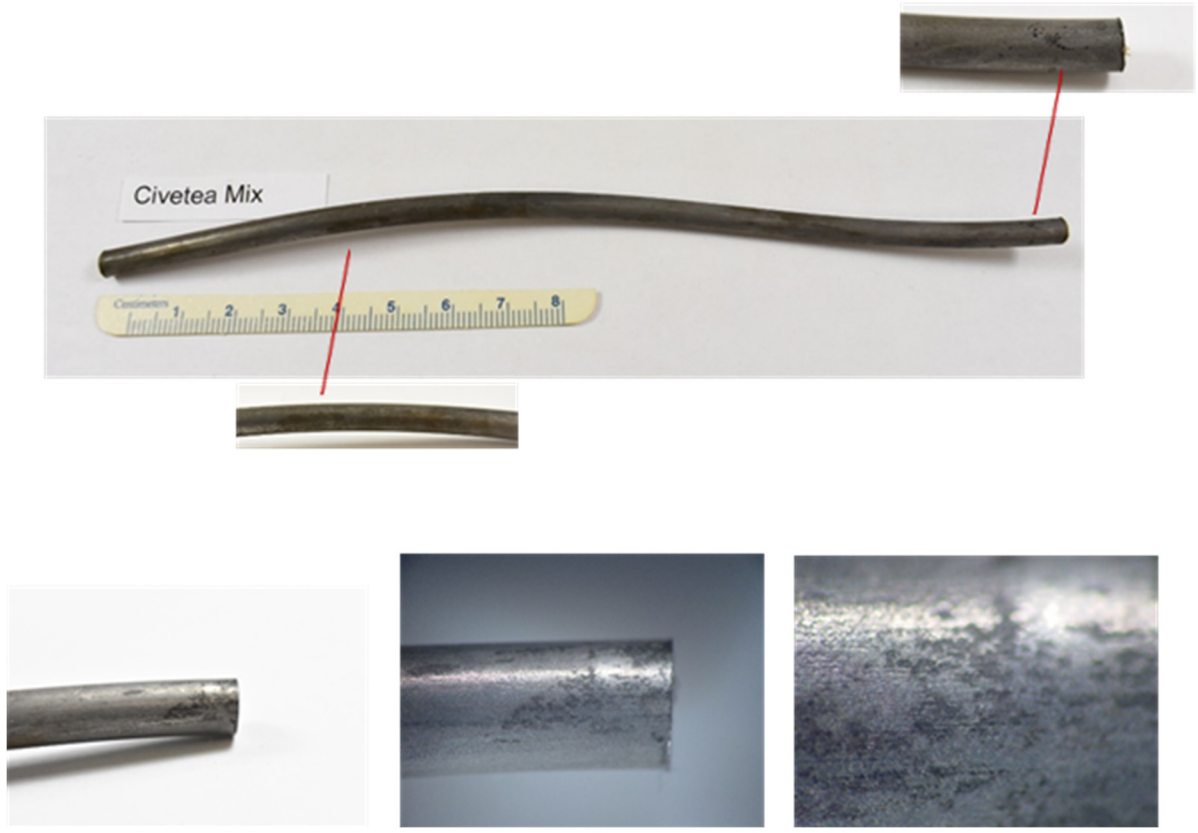


Figure 251. Coated with filler Civetea and sprayed with 1.5 milliliter of solution containing fungi mix (close-up 2).

Figure 252. Coated with filler Sanchem and sprayed with 1.5 milliliter of solution containing fungi mix (close-up).

Figure 253. Coated with filler and sprayed with 1.5 milliliter of solution containing fungus PC

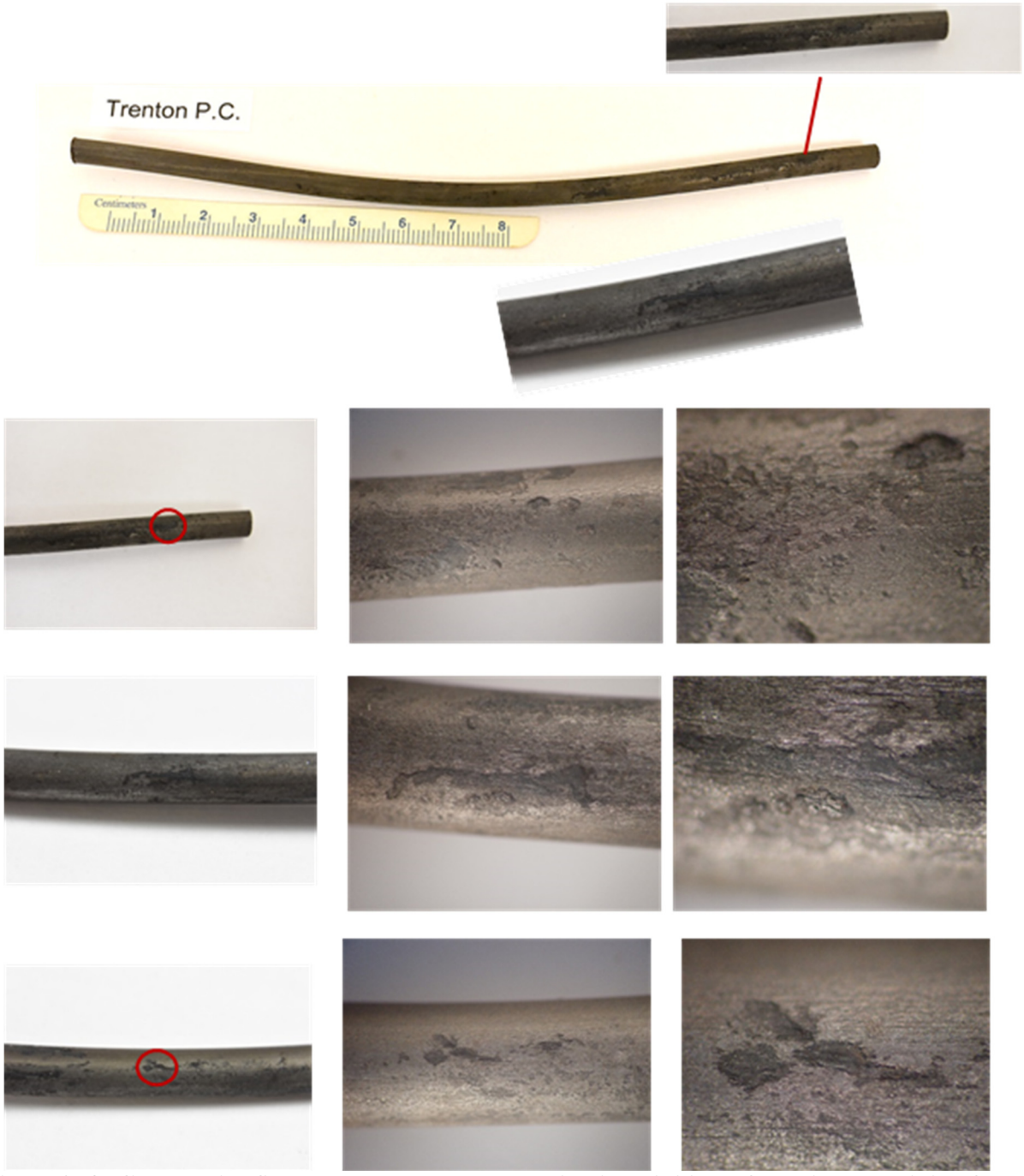


Figure 254. Coated with filler Trenton and sprayed with 1.5 milliliter of solution containing fungus PC (close-up).

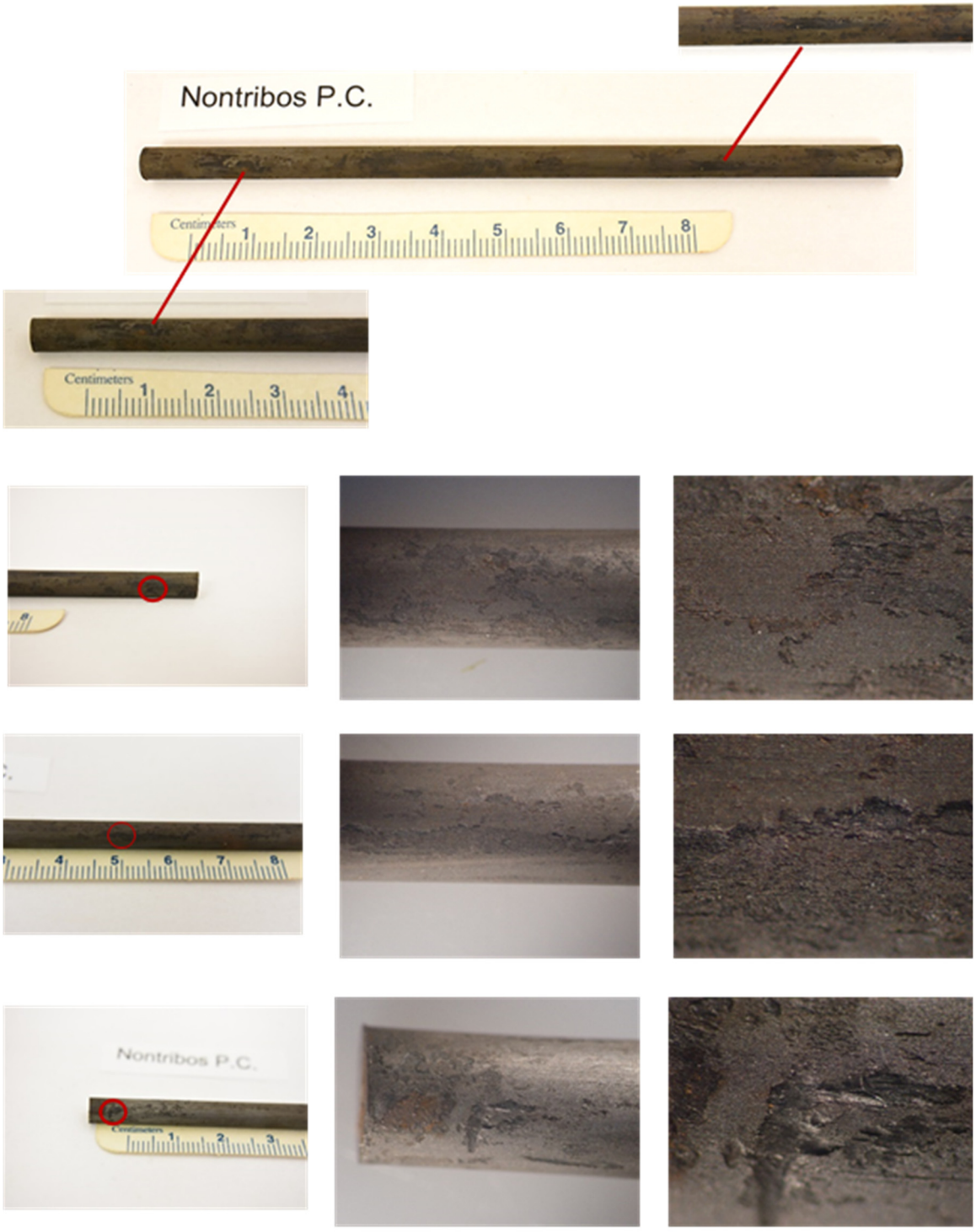


Figure 255. Coated with filler Nontribos and sprayed with 1.5 milliliter of solution containing fungus PC (close-up).

Figure 256. Coated with filler Sonneborn and sprayed with 1.5 milliliter of solution containing fungus PC (close-up).

Figure 257. Coated with filler Civetea and sprayed with 1.5 milliliter of solution containing fungus PC (close-up).

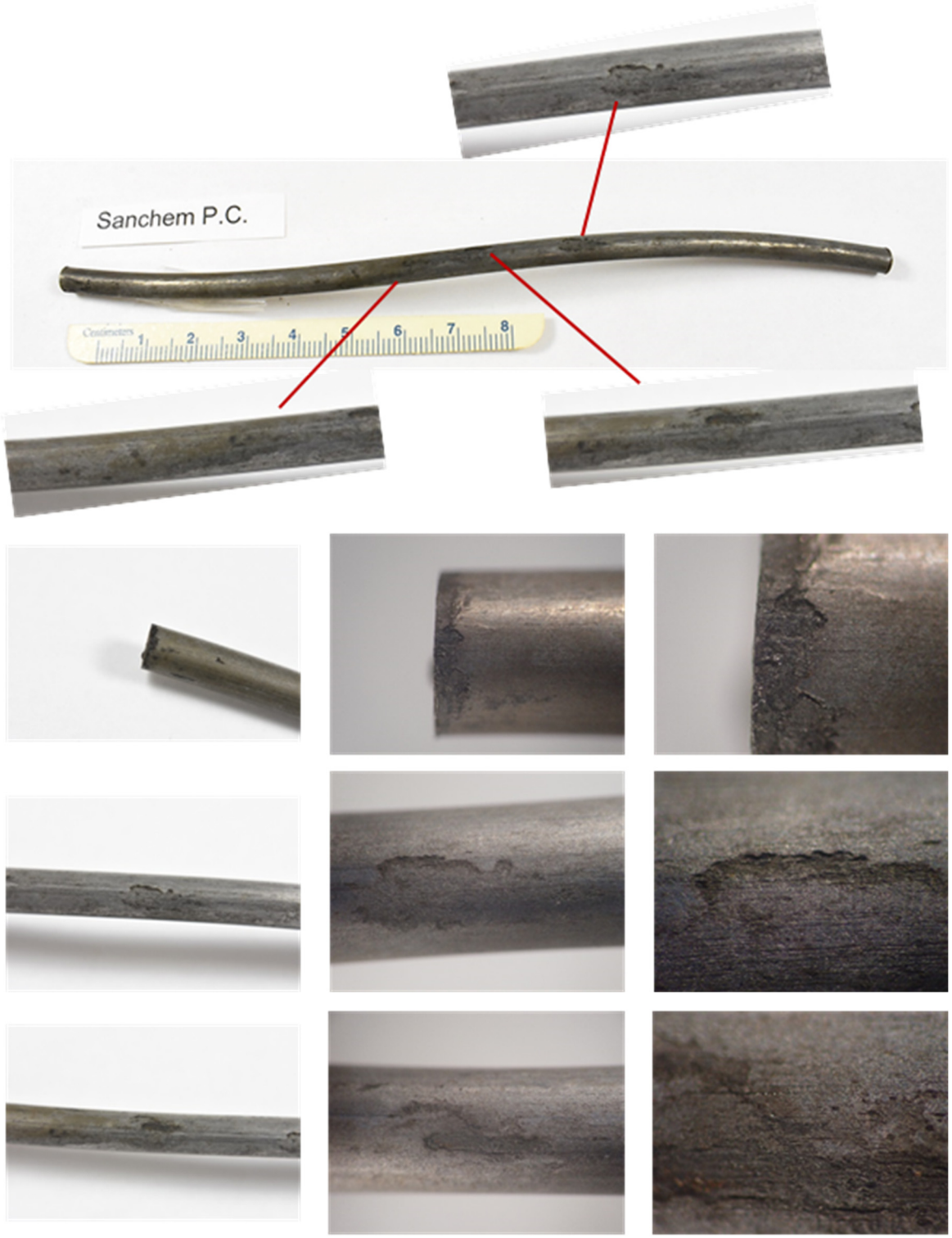


Figure 258. Coated with filler Sanchem and sprayed with 1.5 milliliter of solution containing fungus PC (close-up).

Appendix D

Figure 259. Coated with filler and sprayed with 0.6 milliliter of solution containing fungus AF

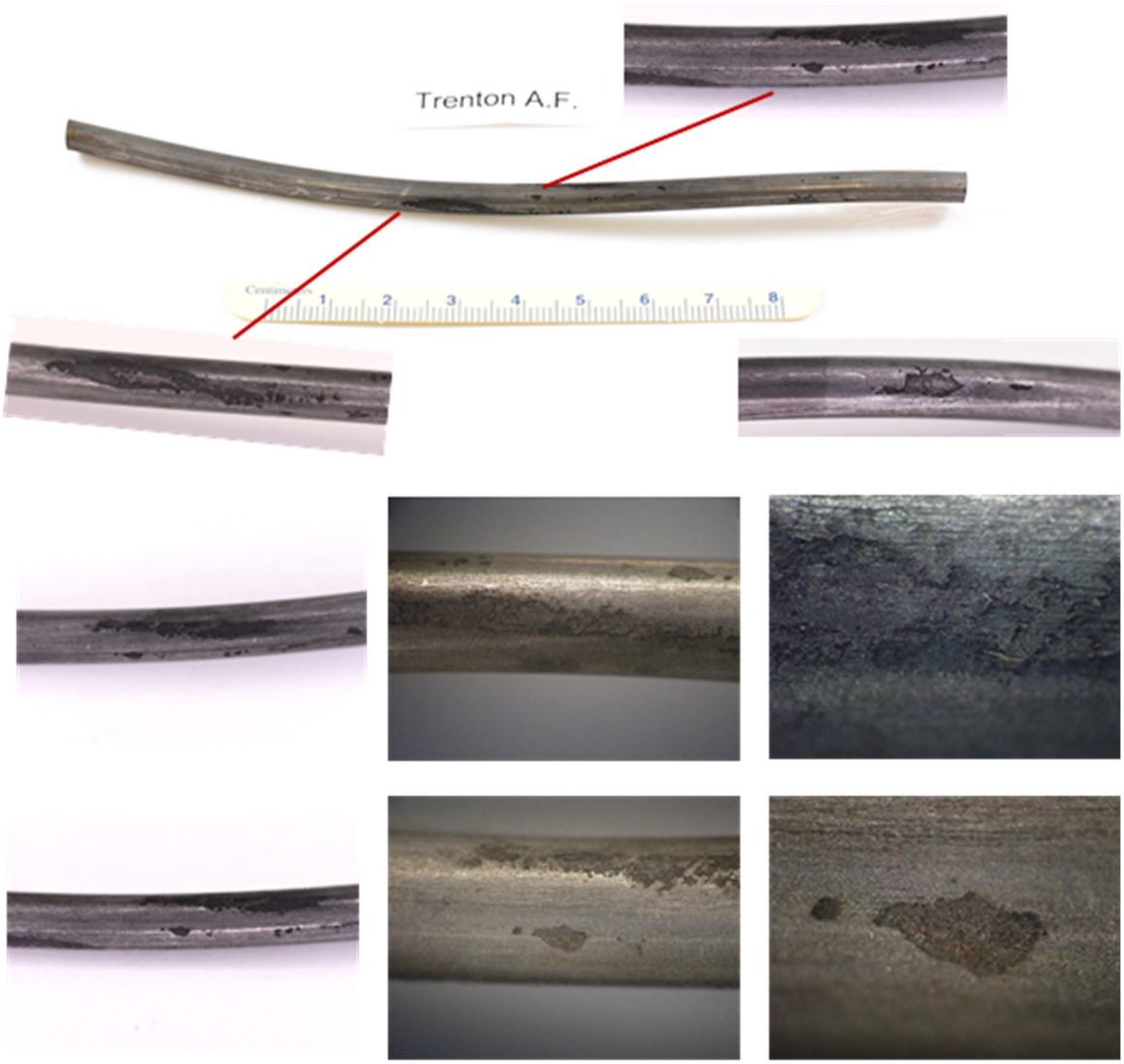


Figure 260. Coated with filler Trenton and sprayed with 0.6 milliliter of solution containing fungus AF (close-up 1).

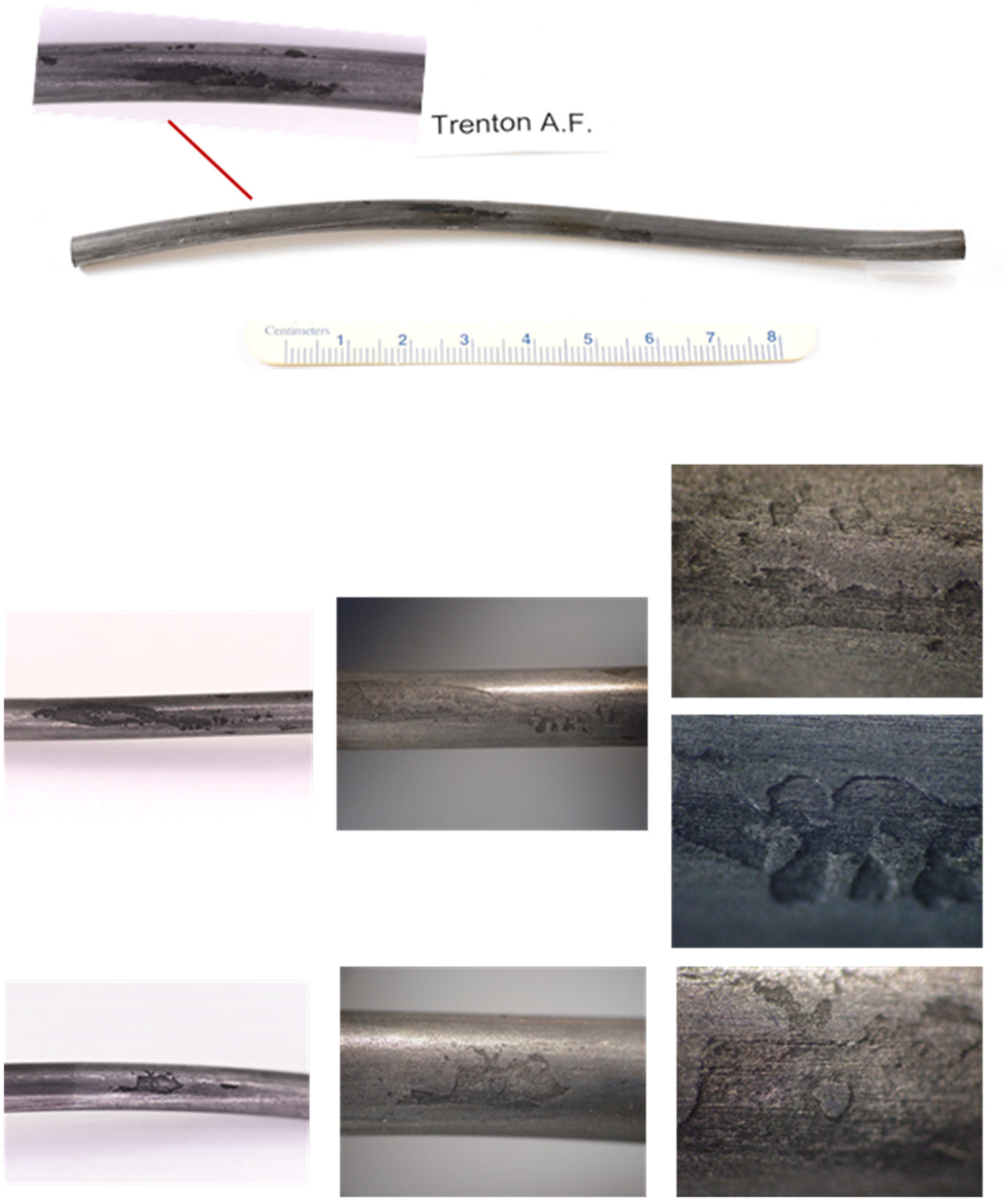


Figure 261. Coated with filler Trenton and sprayed with 0.6 milliliter of solution containing fungus AF (close-up 2).

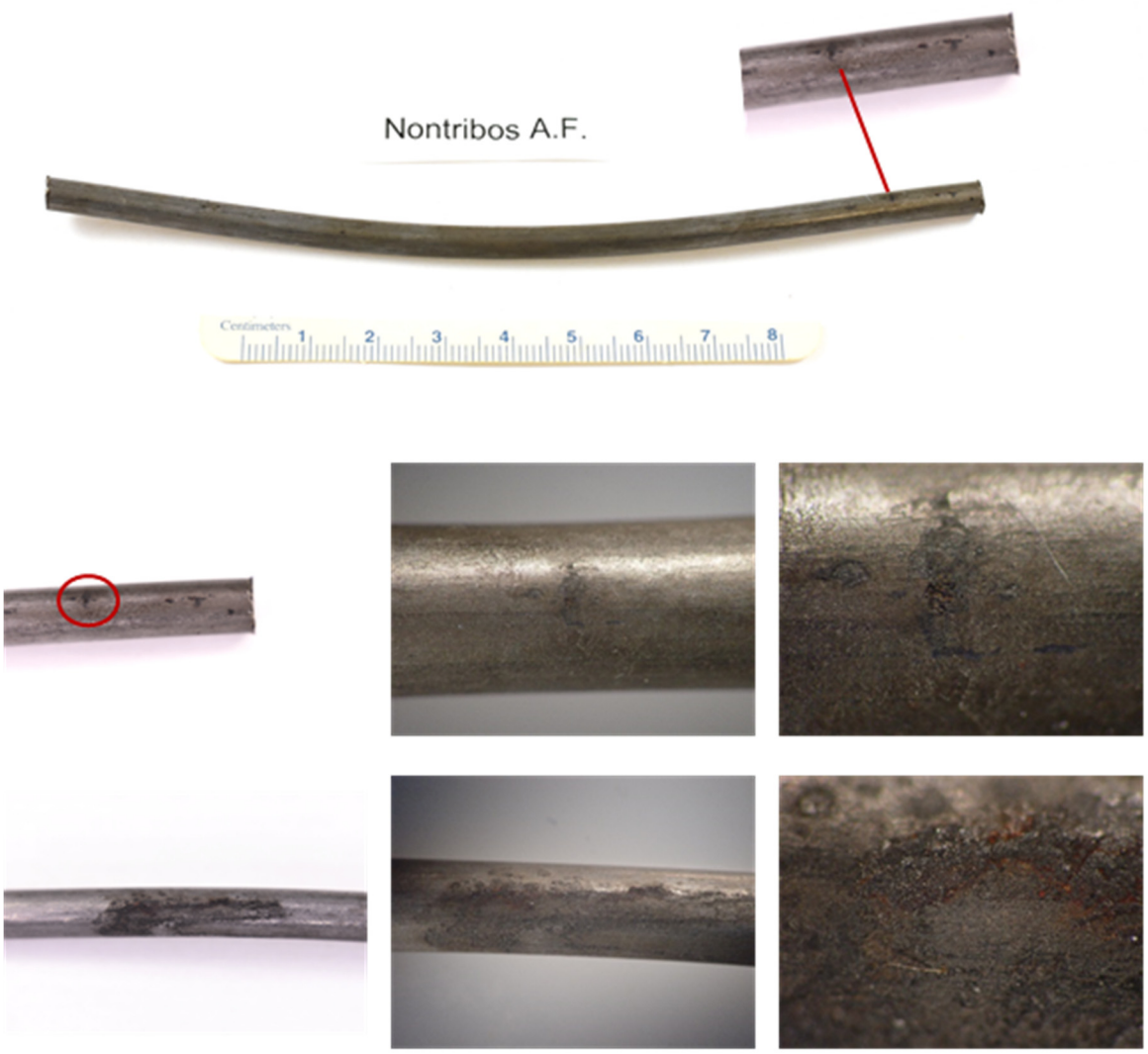


Figure 262. Coated with filler Nontribos and sprayed with 0.6 milliliter of solution containing fungus AF (close-up 1).

Figure 263. Coated with filler Nontribos and sprayed with 0.6 milliliter of solution containing fungus AF (close-up 2).

Figure 264. Coated with filler Sonneborn and sprayed with 0.6 milliliter of solution containing fungus AF (close-p view 1).

Figure 265. Coated with filler Sonneborn and sprayed with 0.6 milliliter of solution containing fungus AF (close-up 4).

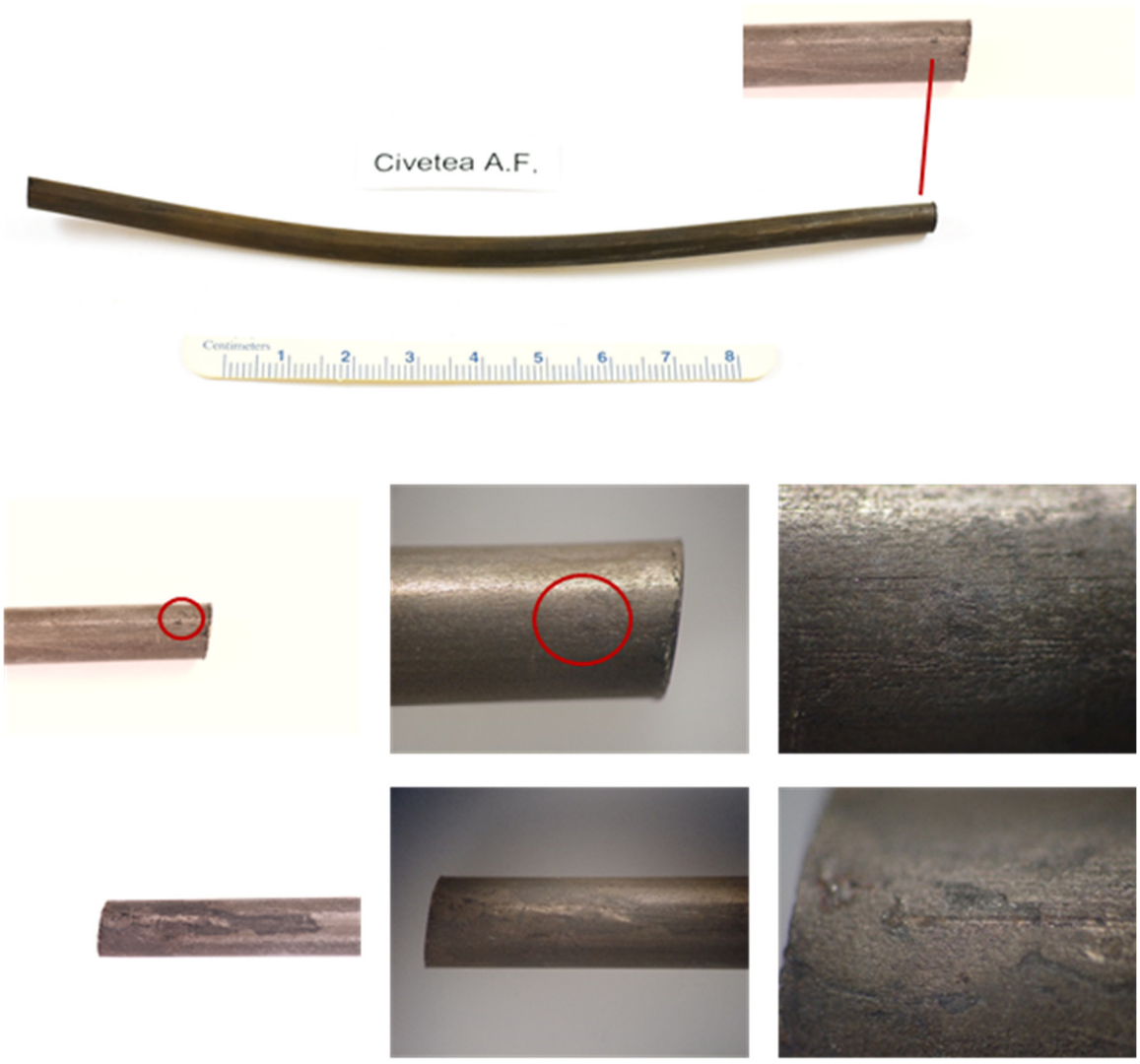


Figure 266. Coated with filler Civetea and sprayed with 0.6 milliliter of solution containing fungus AF (close-up 1).

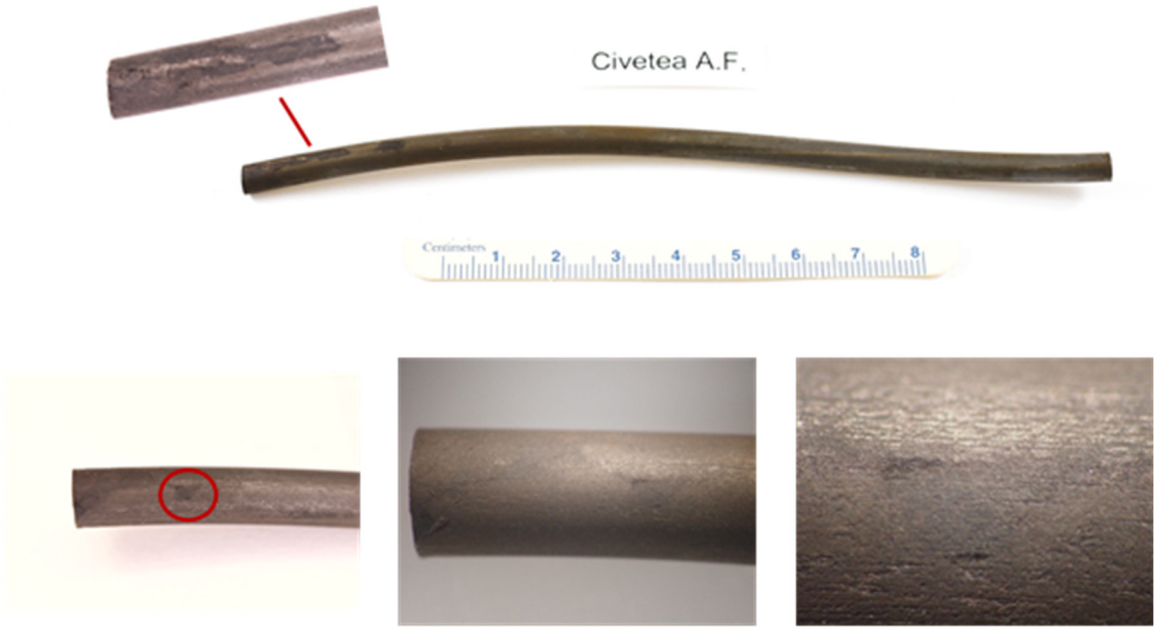


Figure 267. Coated with filler Civetea and sprayed with 0.6 milliliter of solution containing fungus AF (close-up 2).

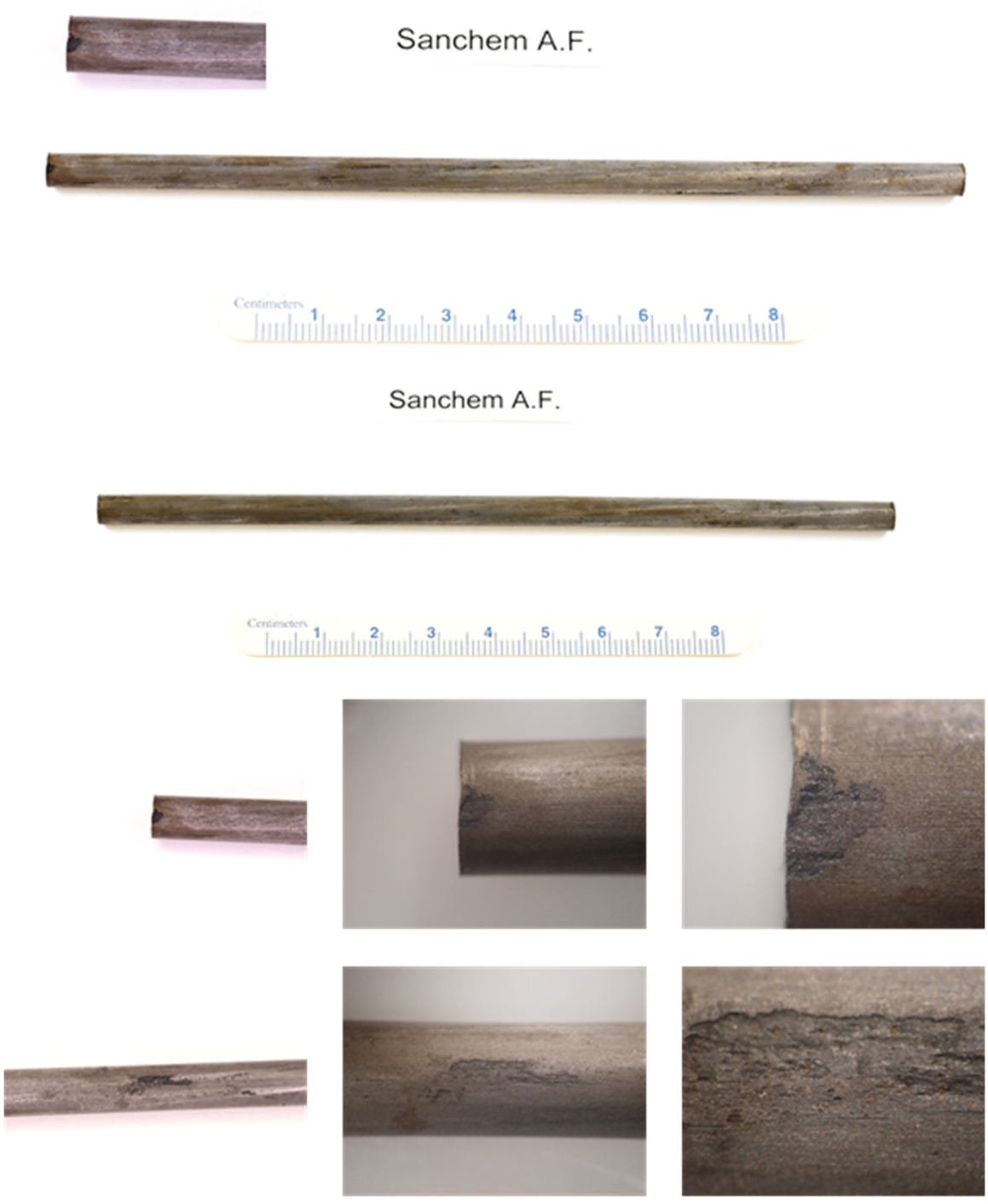


Figure 268. Coated with filler Sanchem and sprayed with 0.6 milliliter of solution containing fungus AF (close-up).

Figure 269. Coated with filler and sprayed with 0.6 milliliter of solution containing fungus FO

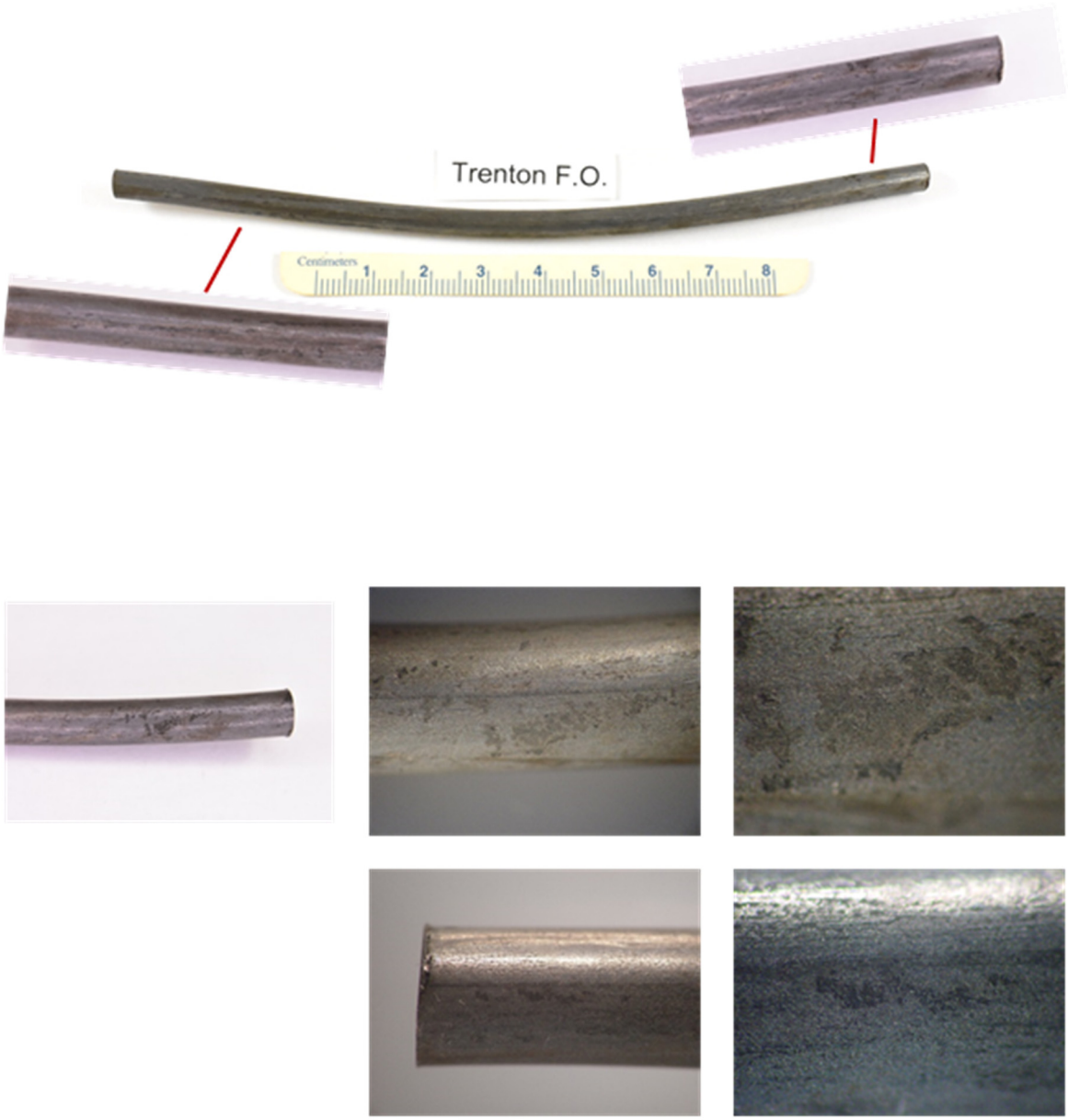


Figure 270. Coated with filler Trenton and sprayed with 0.6 milliliter of solution containing fungus FO (close-up 1).

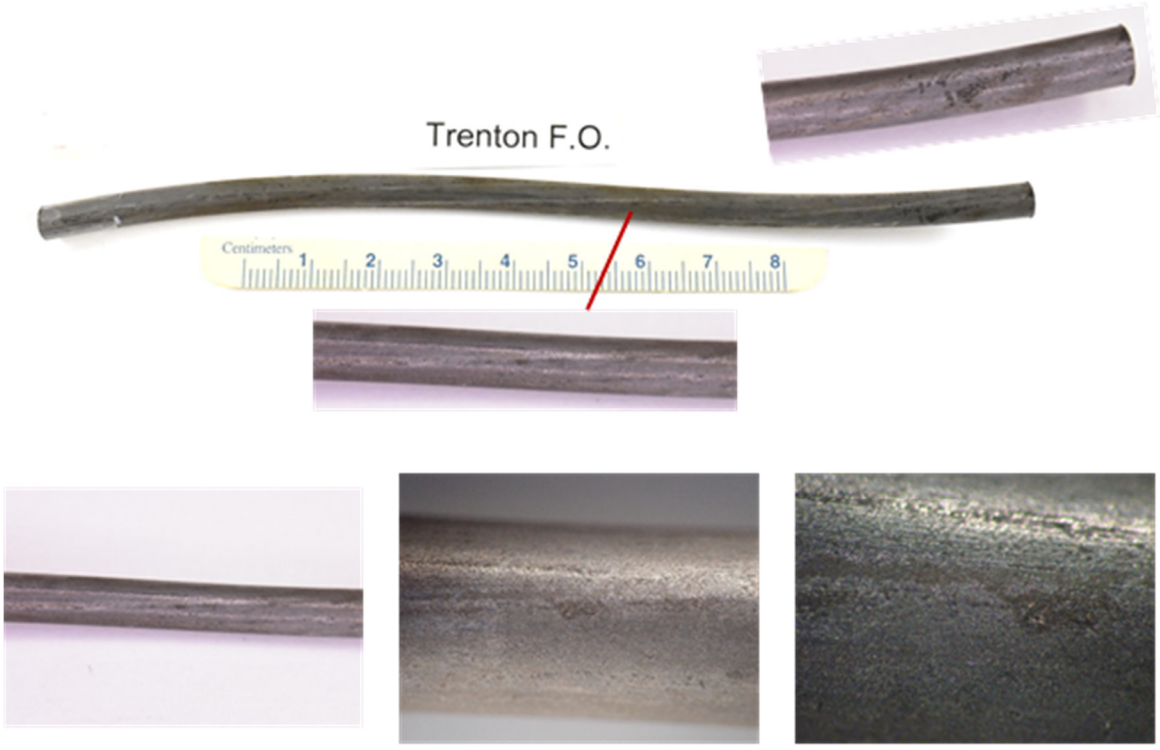


Figure 271. Coated with filler Trenton and sprayed with 0.6 milliliter of solution containing fungus FO (close-up 2).

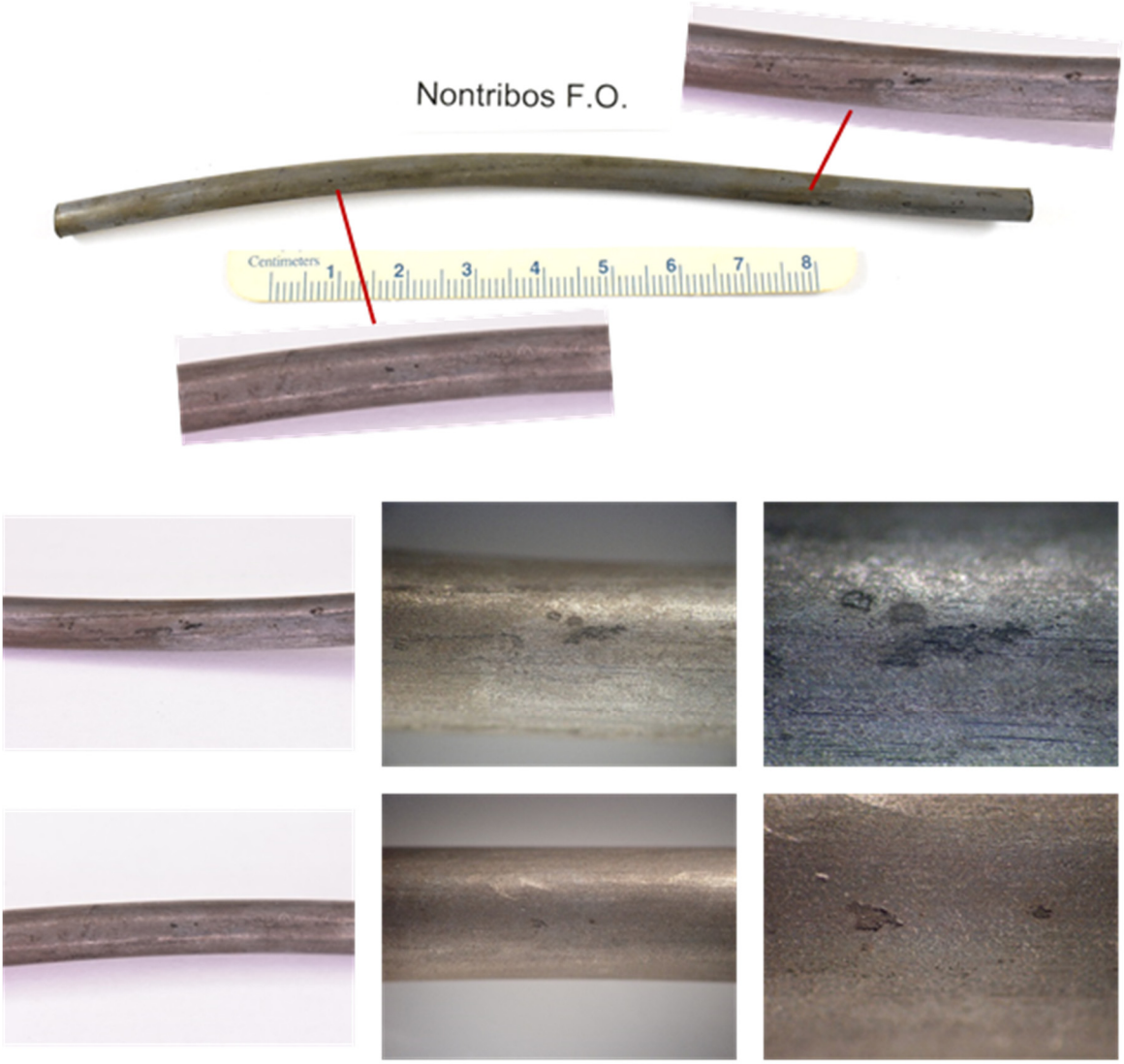


Figure 272. Coated with filler Nontribos and sprayed with 0.6 milliliter of solution containing fungus FO (close-up 1).

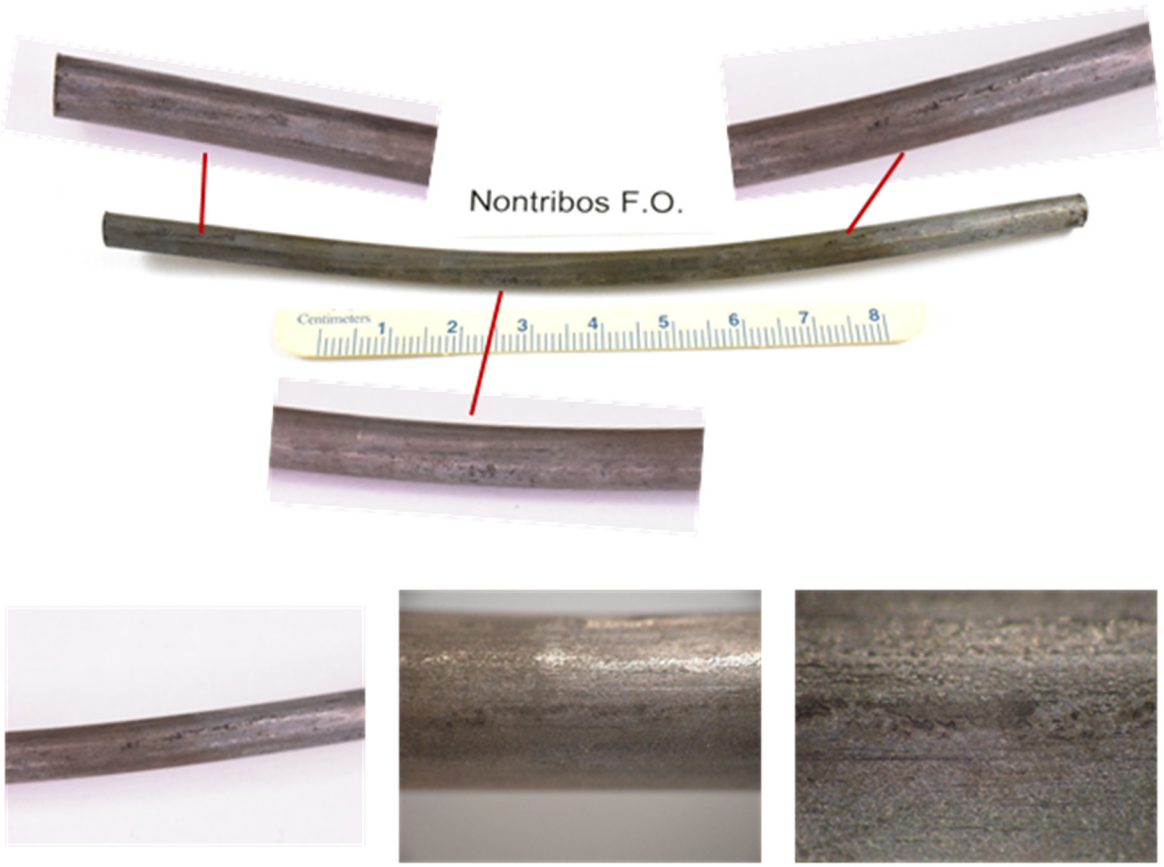


Figure 273. Coated with filler Nontribos and sprayed with 0.6 milliliter of solution containing fungus FO (close-up 2).

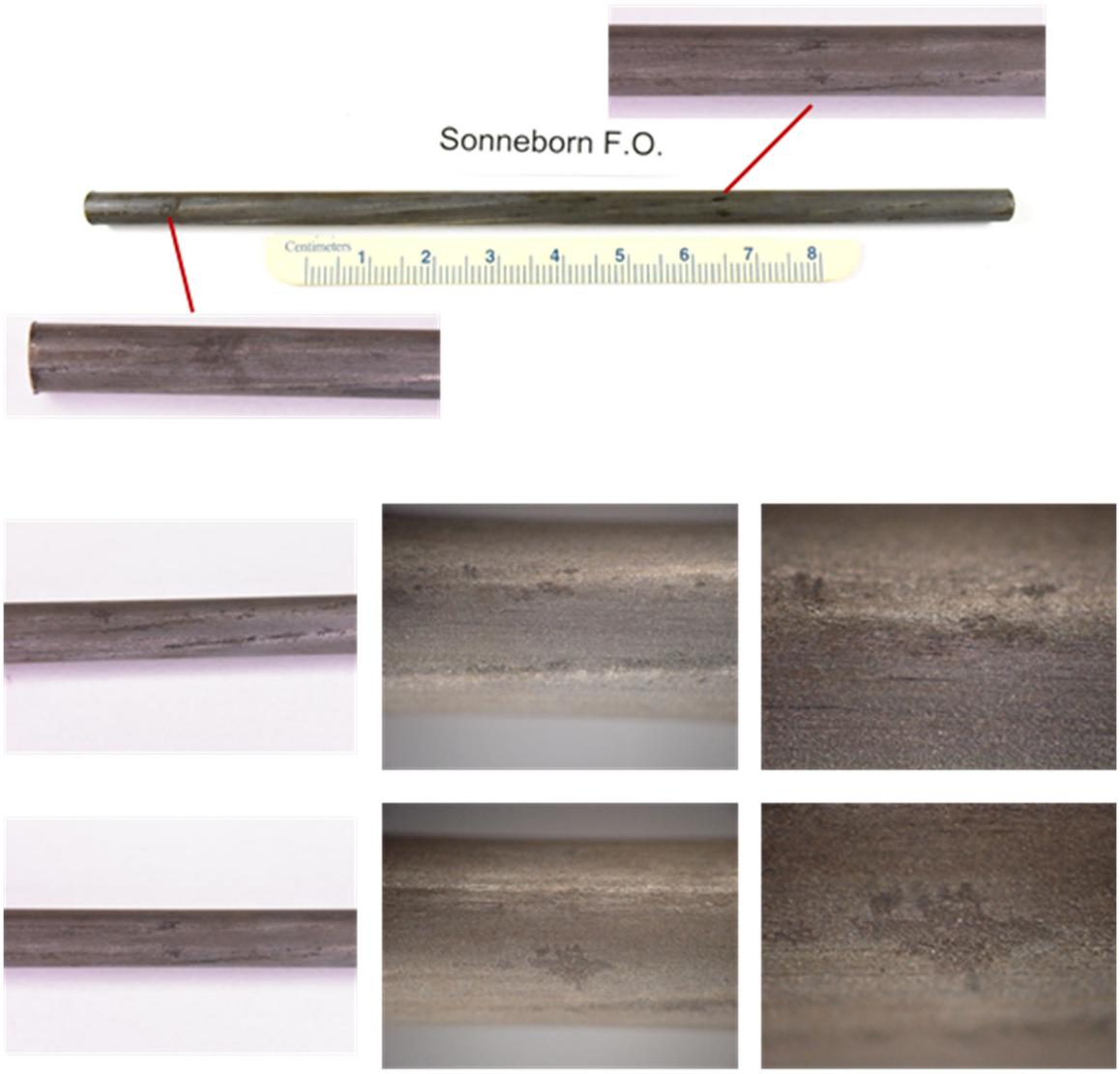


Figure 274. Coated with filler Sonneborn and sprayed with 0.6 milliliter of solution containing fungus FO (close-up 1).

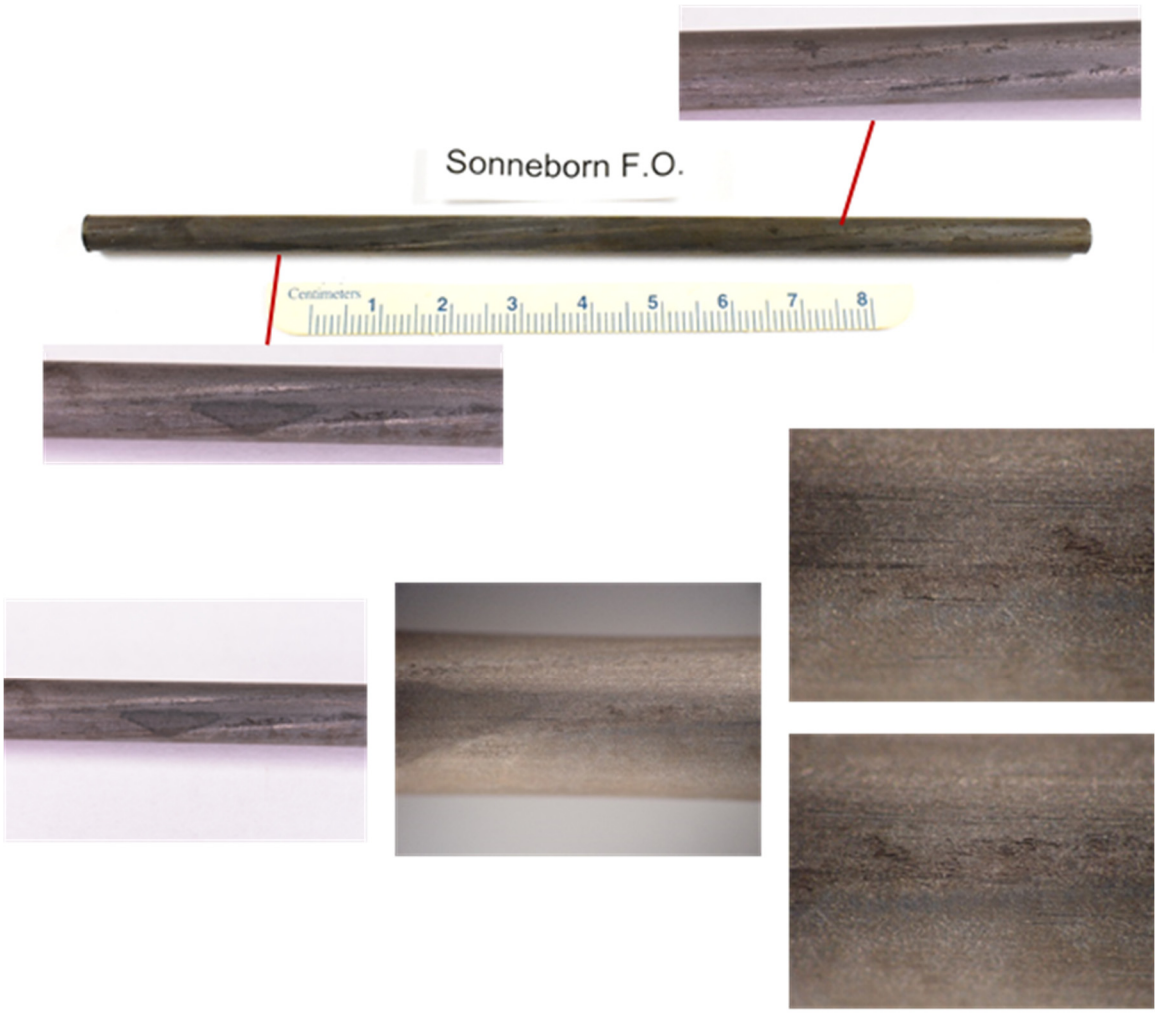


Figure 275. Coated with filler Sonneborn and sprayed with 0.6 milliliter of solution containing fungus FO (close-up 2).

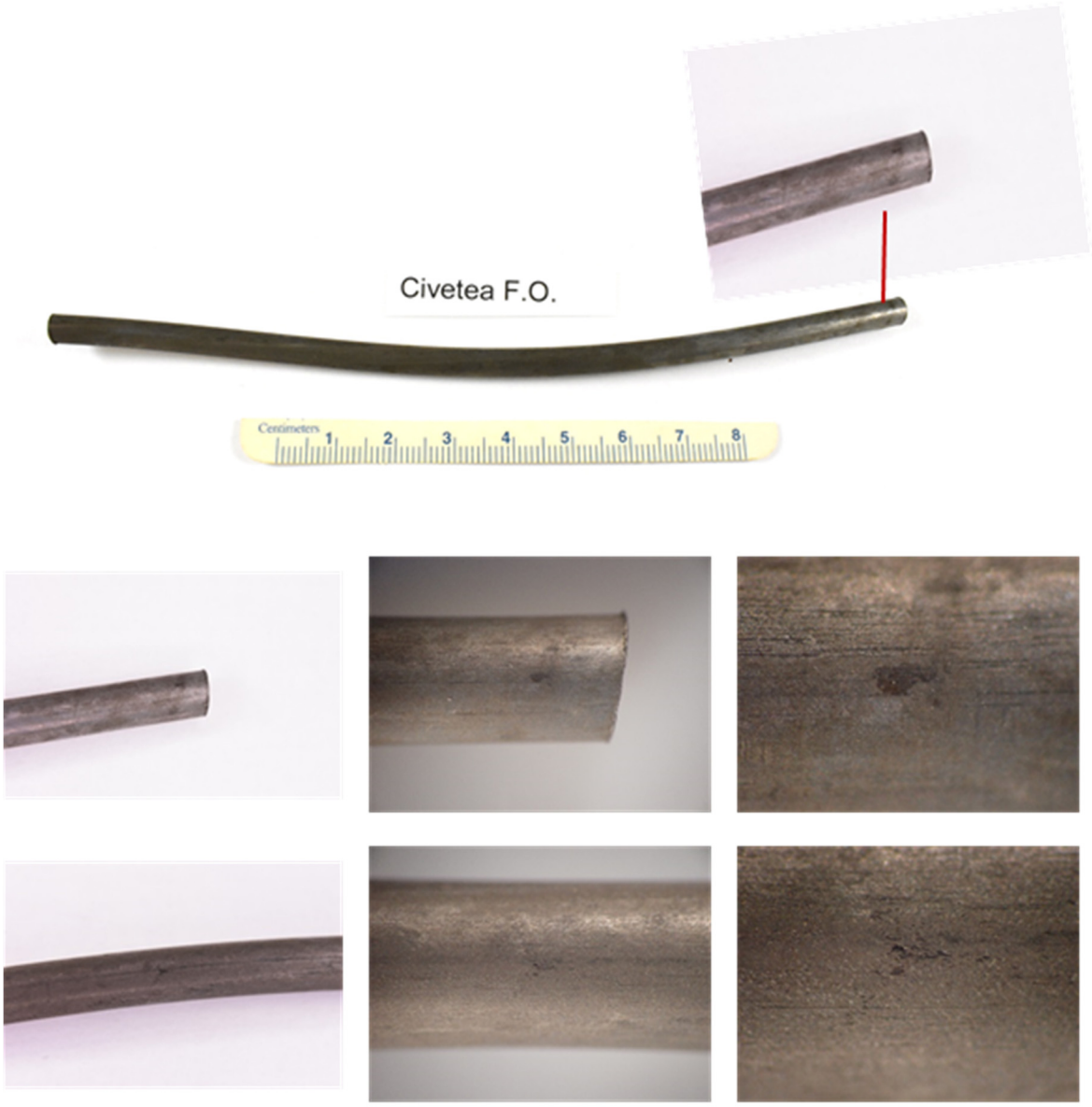


Figure 276. Coated with filler Civetea and sprayed with 0.6 milliliter of solution containing fungus FO (close-up 2).

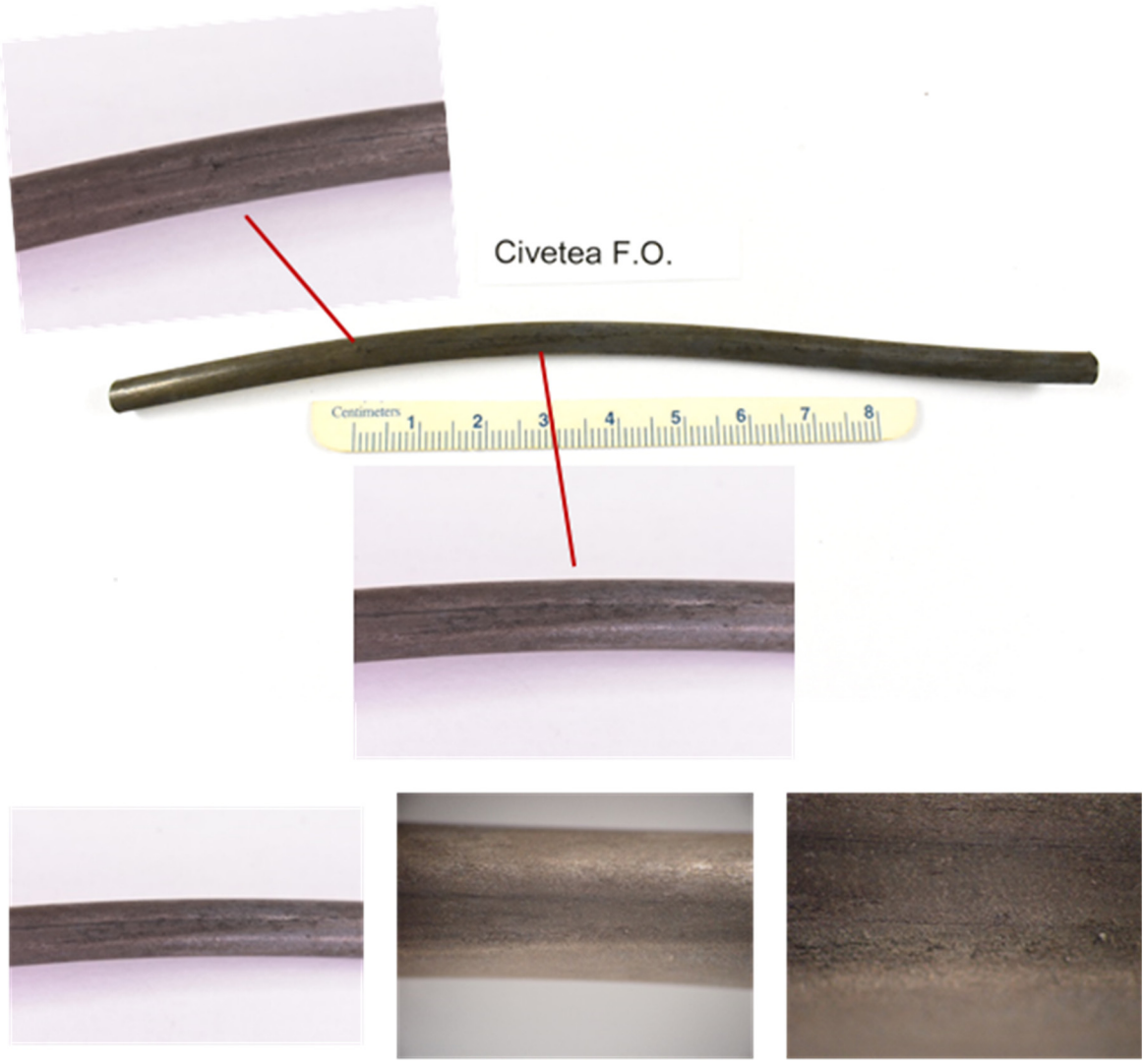


Figure 277. Coated with filler Civetea and sprayed with 0.6 milliliter of solution containing fungus FO (close-up 1).

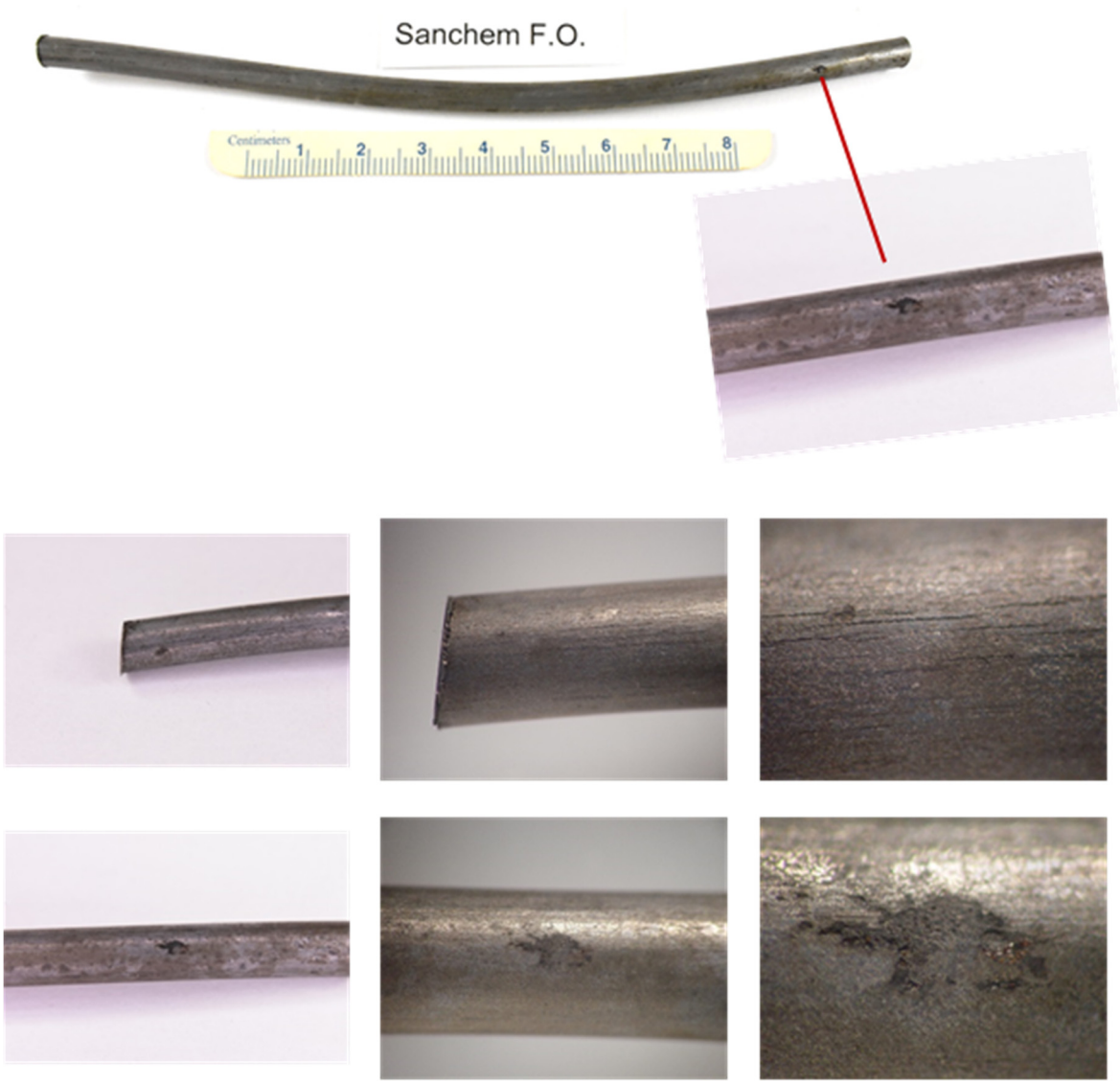


Figure 278. Coated with filler Sanchem and sprayed with 0.6 milliliter of solution containing fungus FO

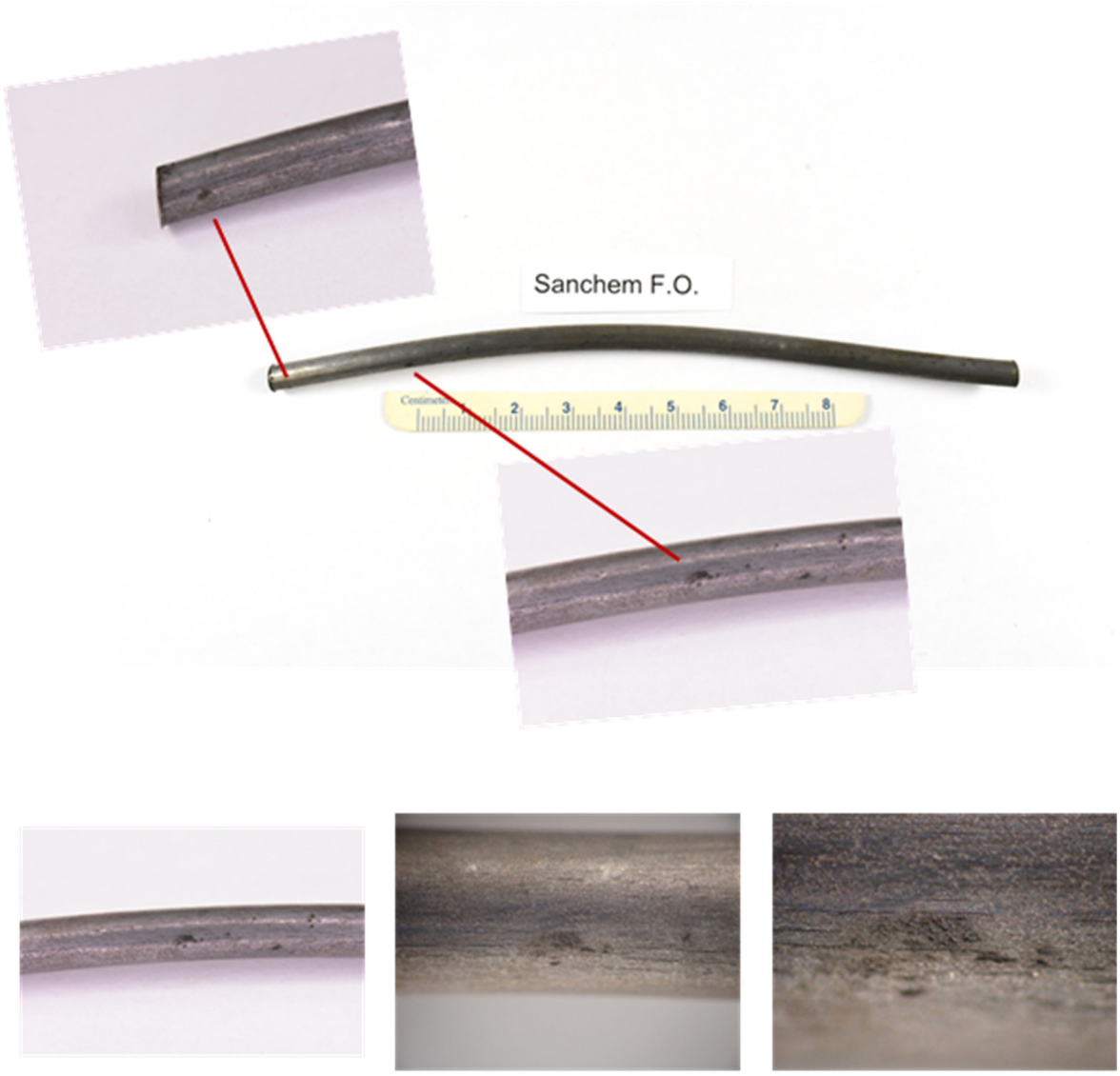


Figure 279. Coated with filler Sanchem and sprayed with 0.6 milliliter of solution containing fungus FO (close-up 2).

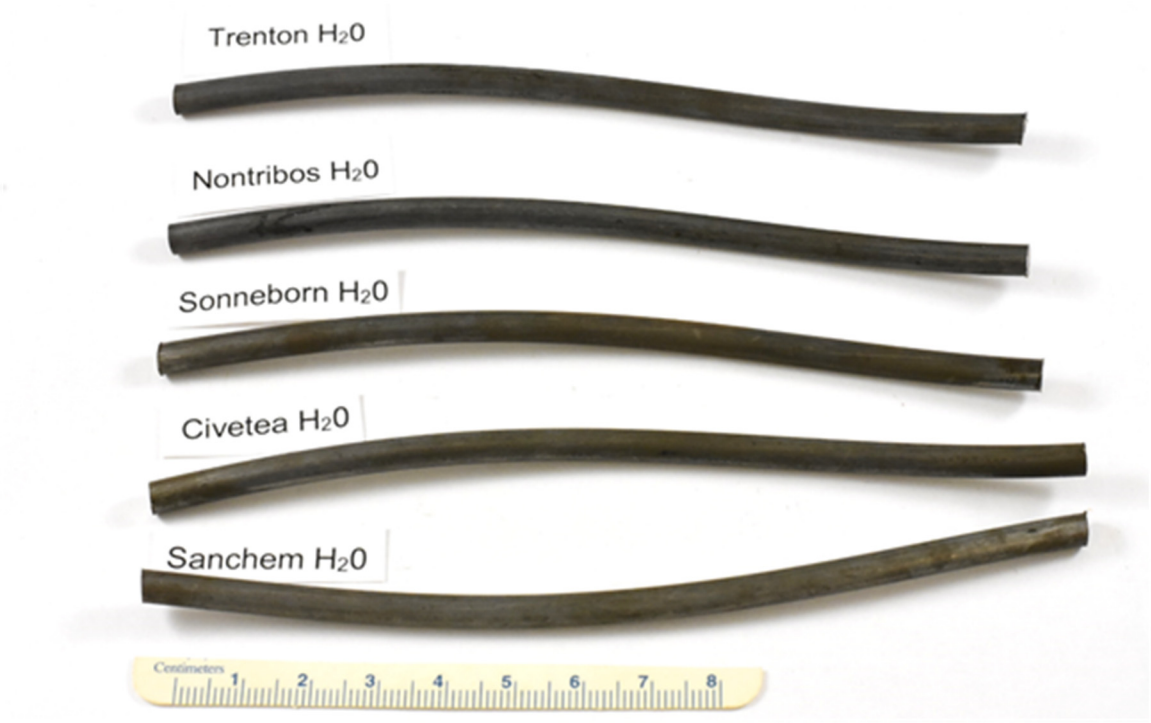


Figure 280. Coated with filler and sprayed with 0.6 milliliter of water.

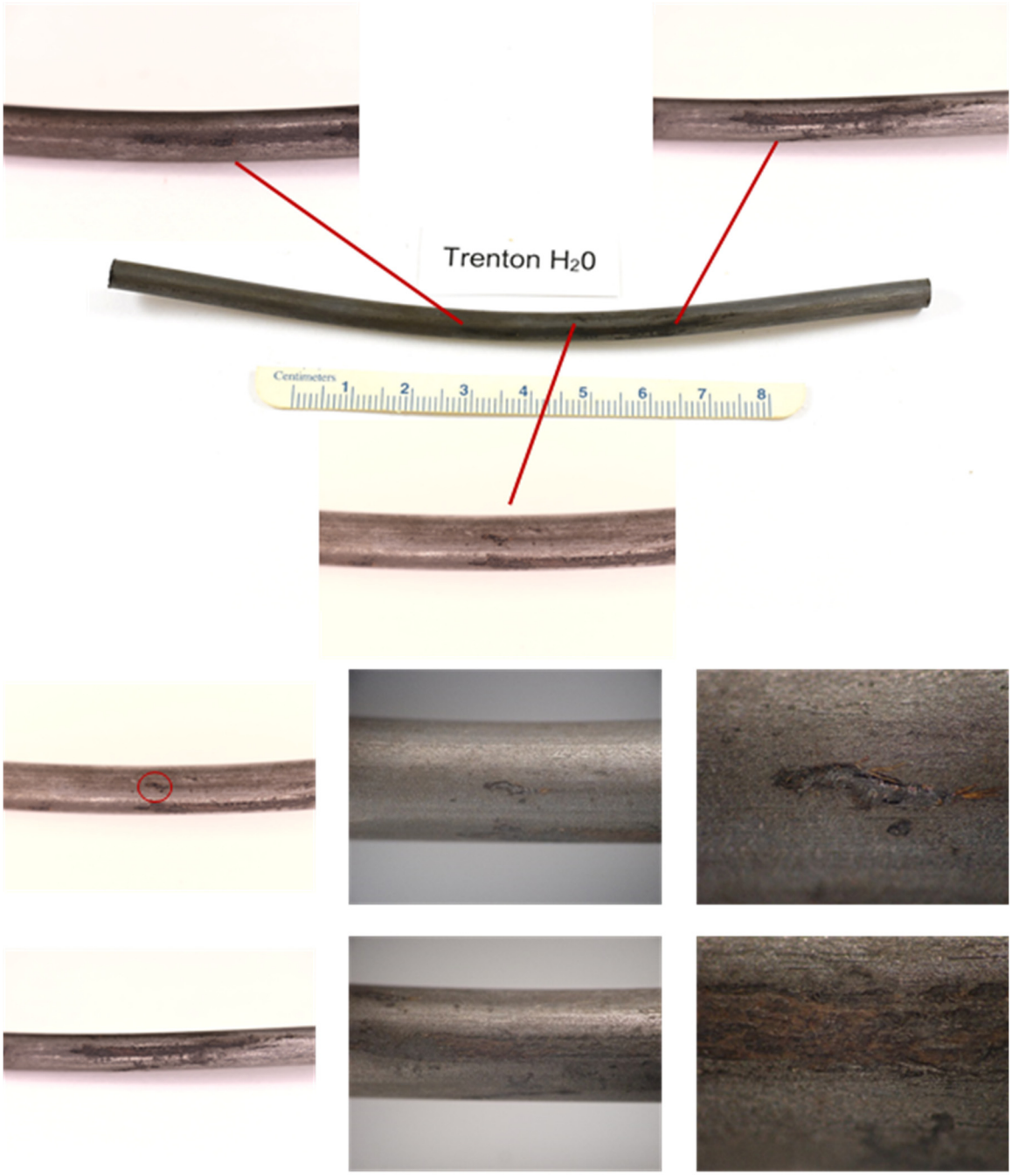


Figure 281. Coated with filler Trenton and sprayed with 0.6 milliliter of water .

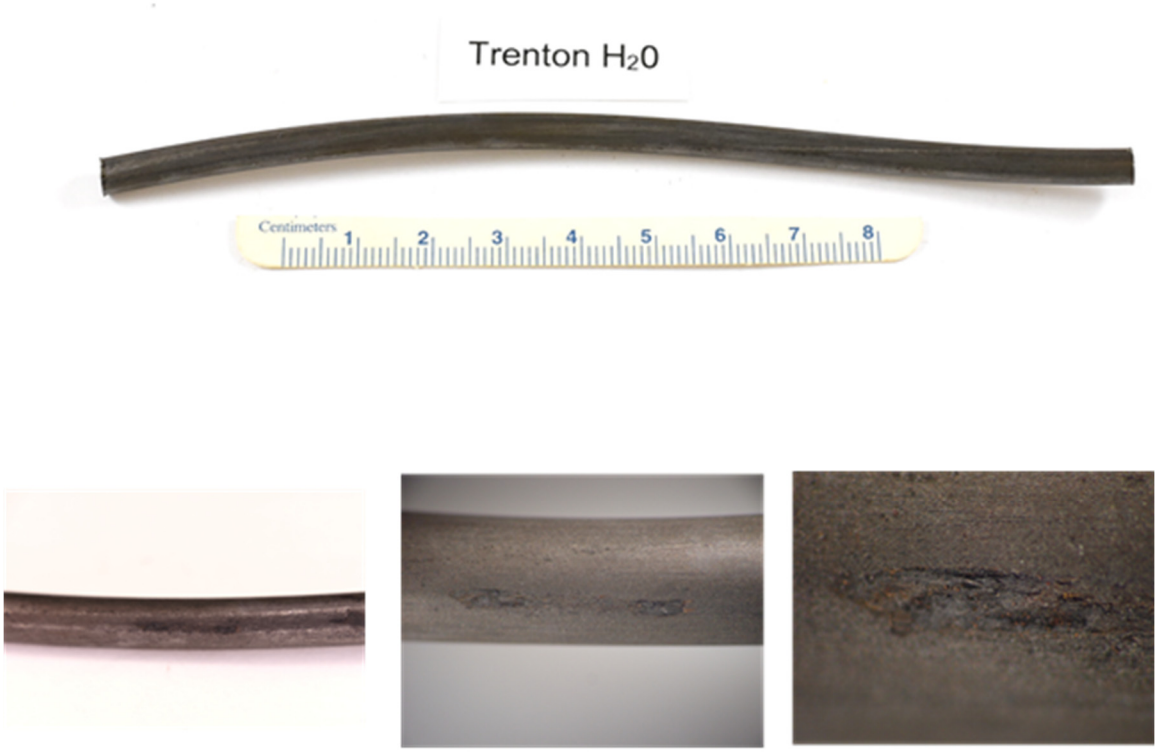


Figure 282. Coated with filler Trenton and sprayed with 0.6 milliliter of water (full view).

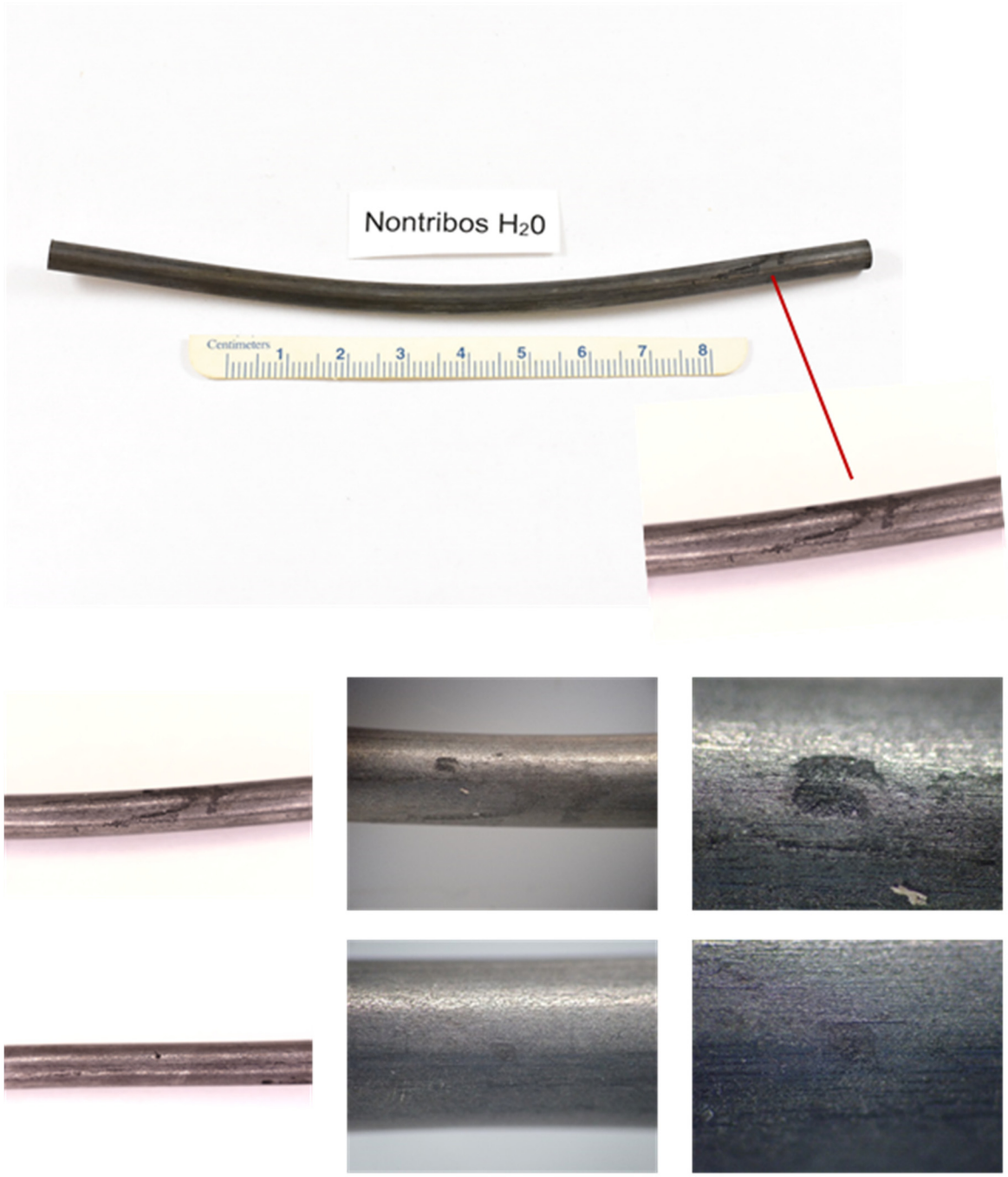


Figure 283. Coated with filler Nontribos and sprayed with 0.6 milliliter of water (close-up).

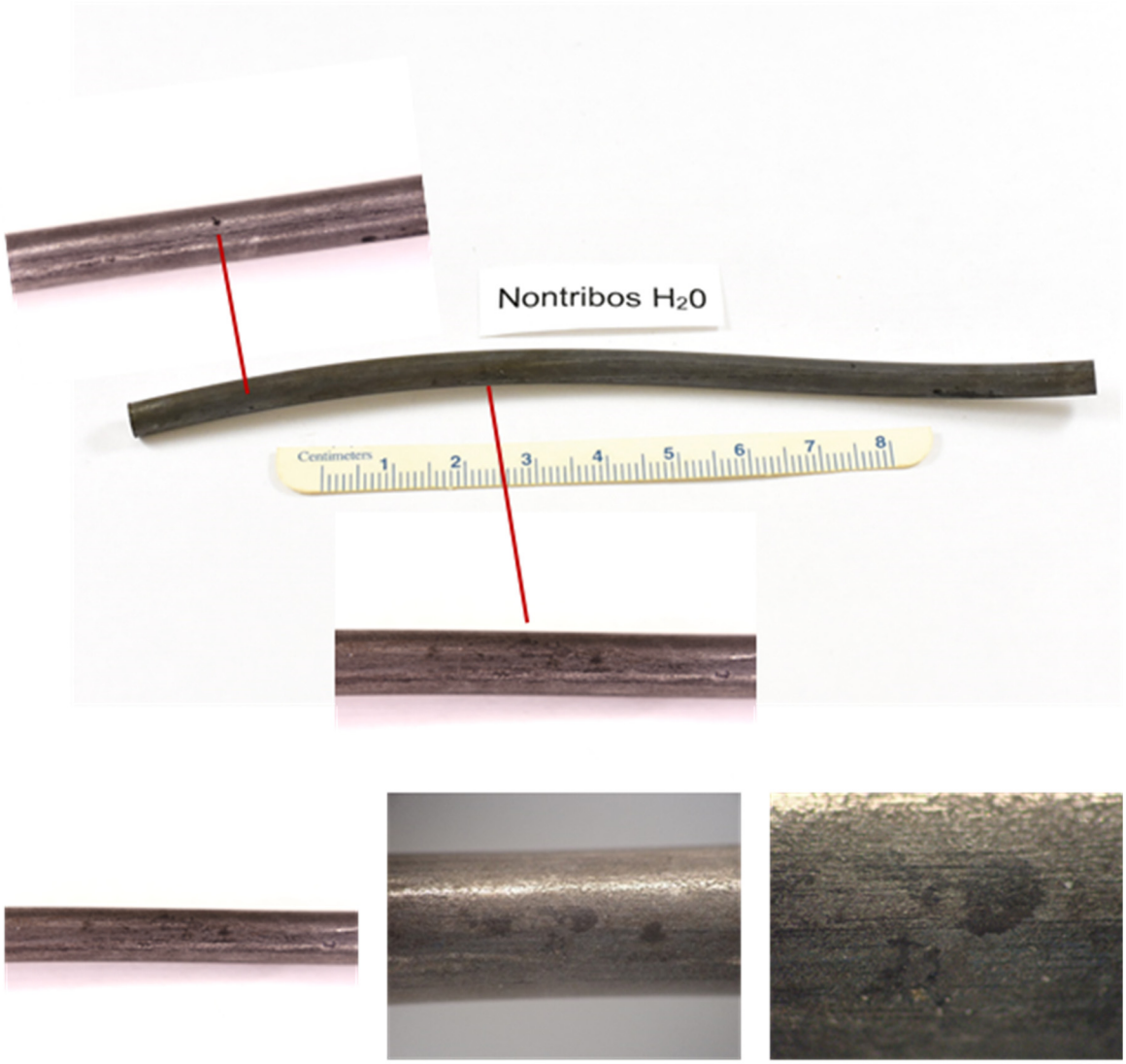


Figure 284. Coated with filler Nontribos and sprayed with 0.6 milliliter of water (close-up 2).

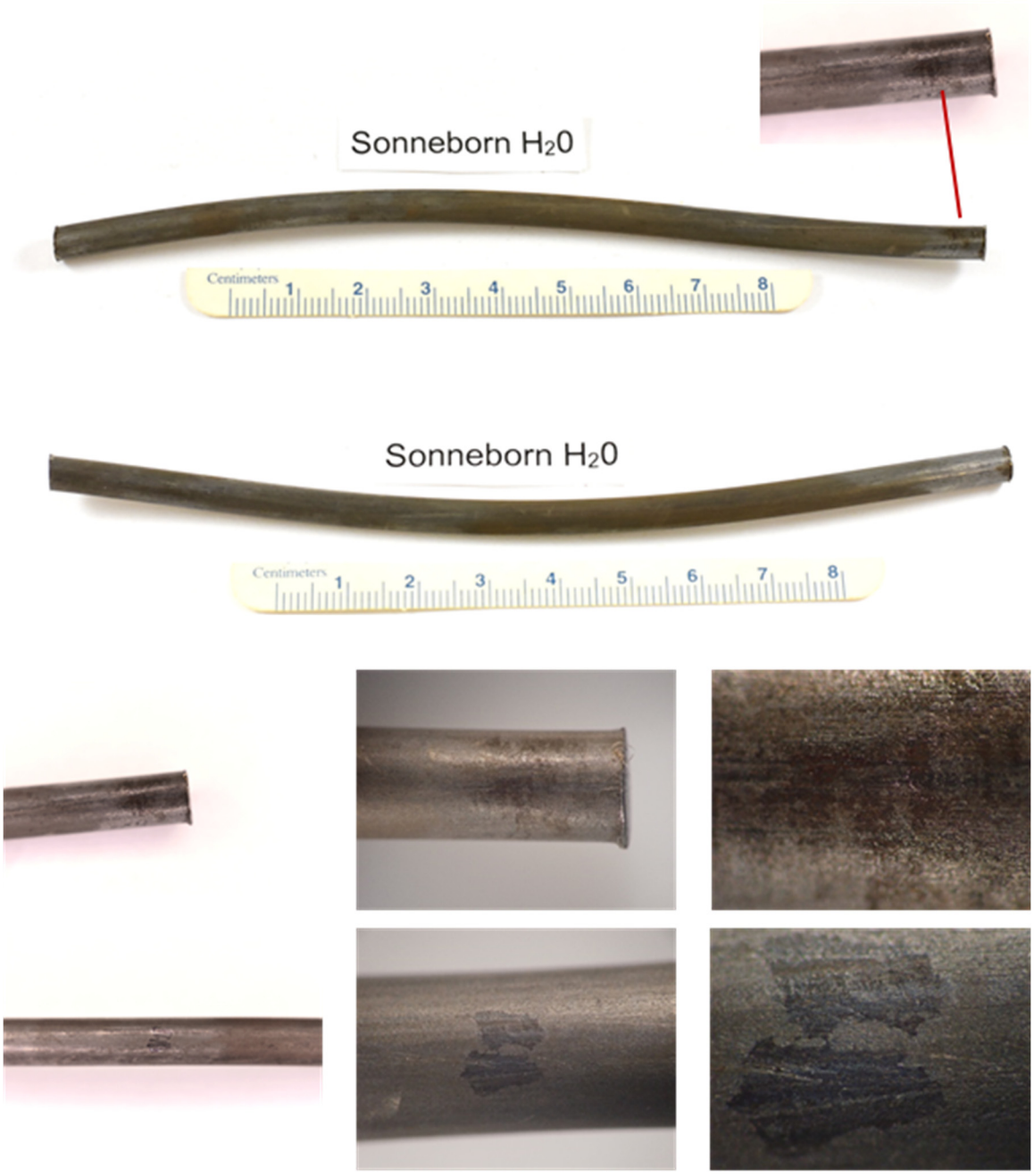


Figure 285. Coated with filler Sonneborn and sprayed with 0.6 milliliter of water (close-up 1).

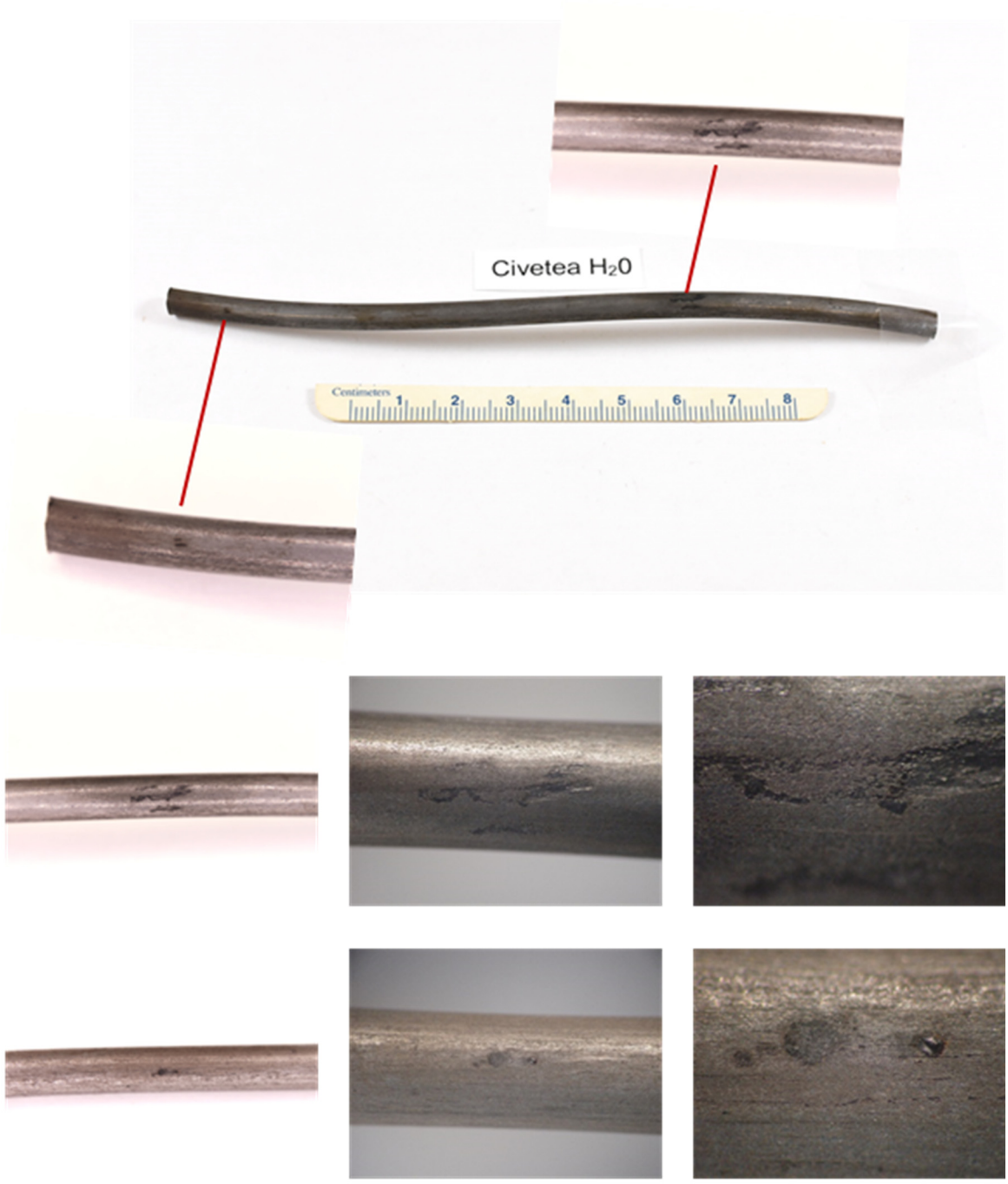


Figure 286. Coated with filler Civetea and sprayed with 0.6 milliliter of water (close-up 1)

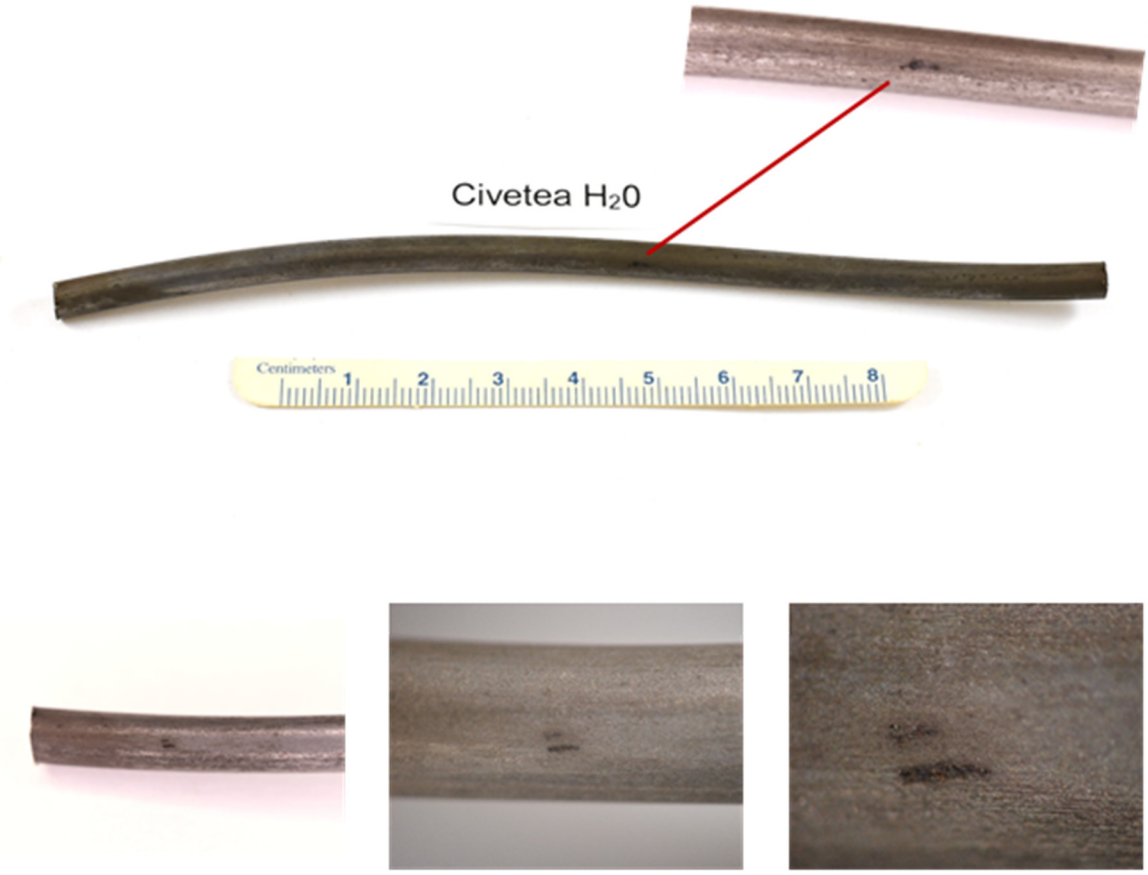


Figure 287. Coated with filler Civetea and sprayed with 0.6 milliliter of water (close-up 2)

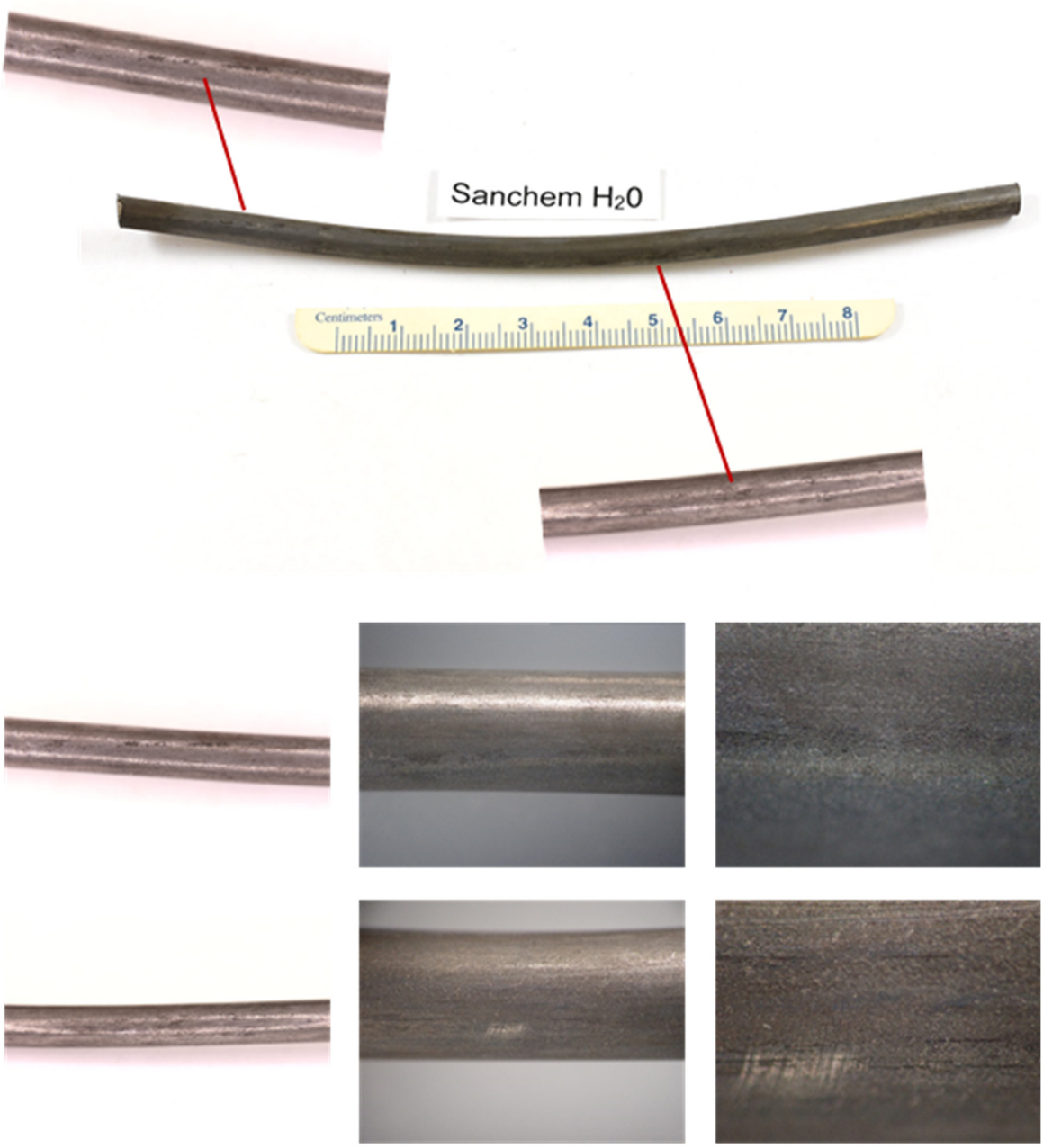


Figure 288. Coated with filler Sanchem and sprayed with 0.6 milliliter of water (close-up 1).

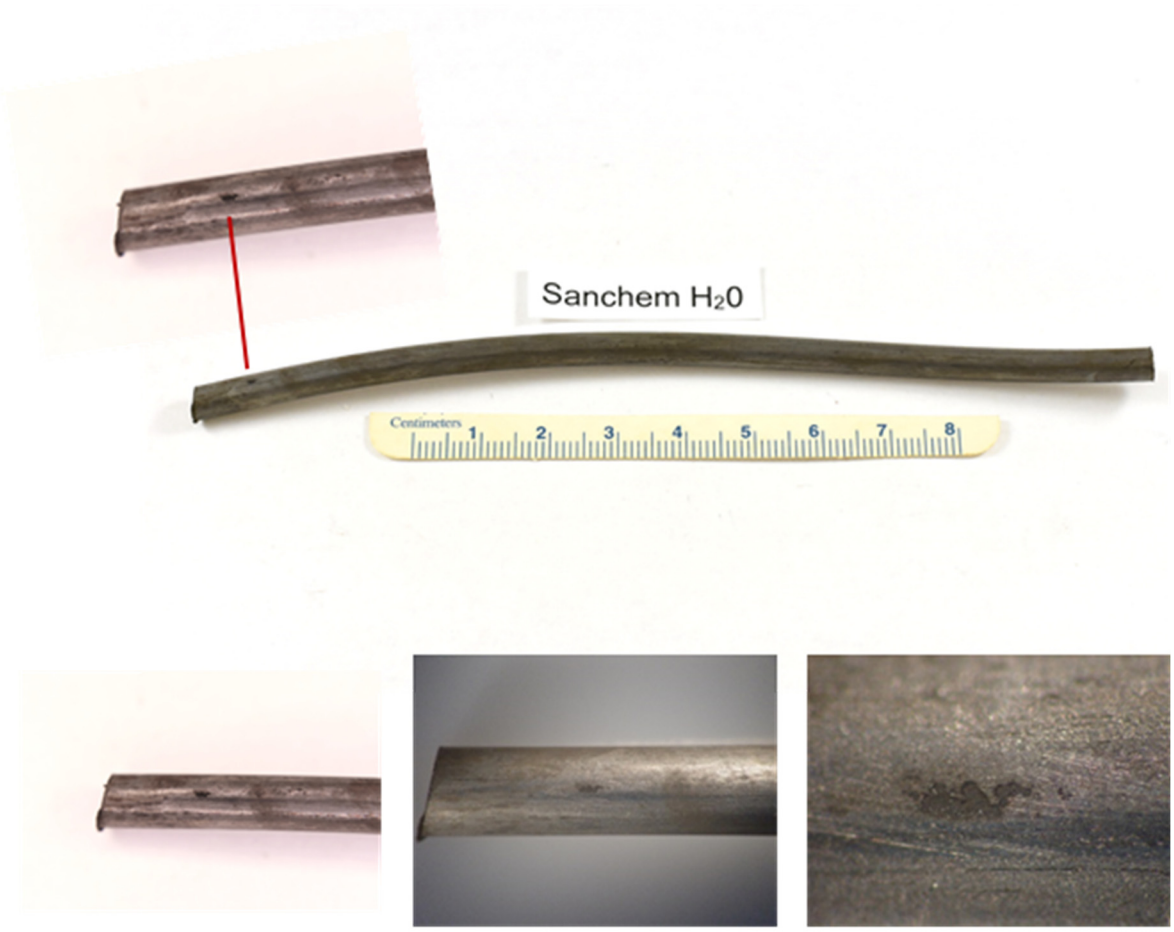


Figure 289. Coated with filler Sanchem and sprayed with 0.6 milliliter of water (close-up 2).

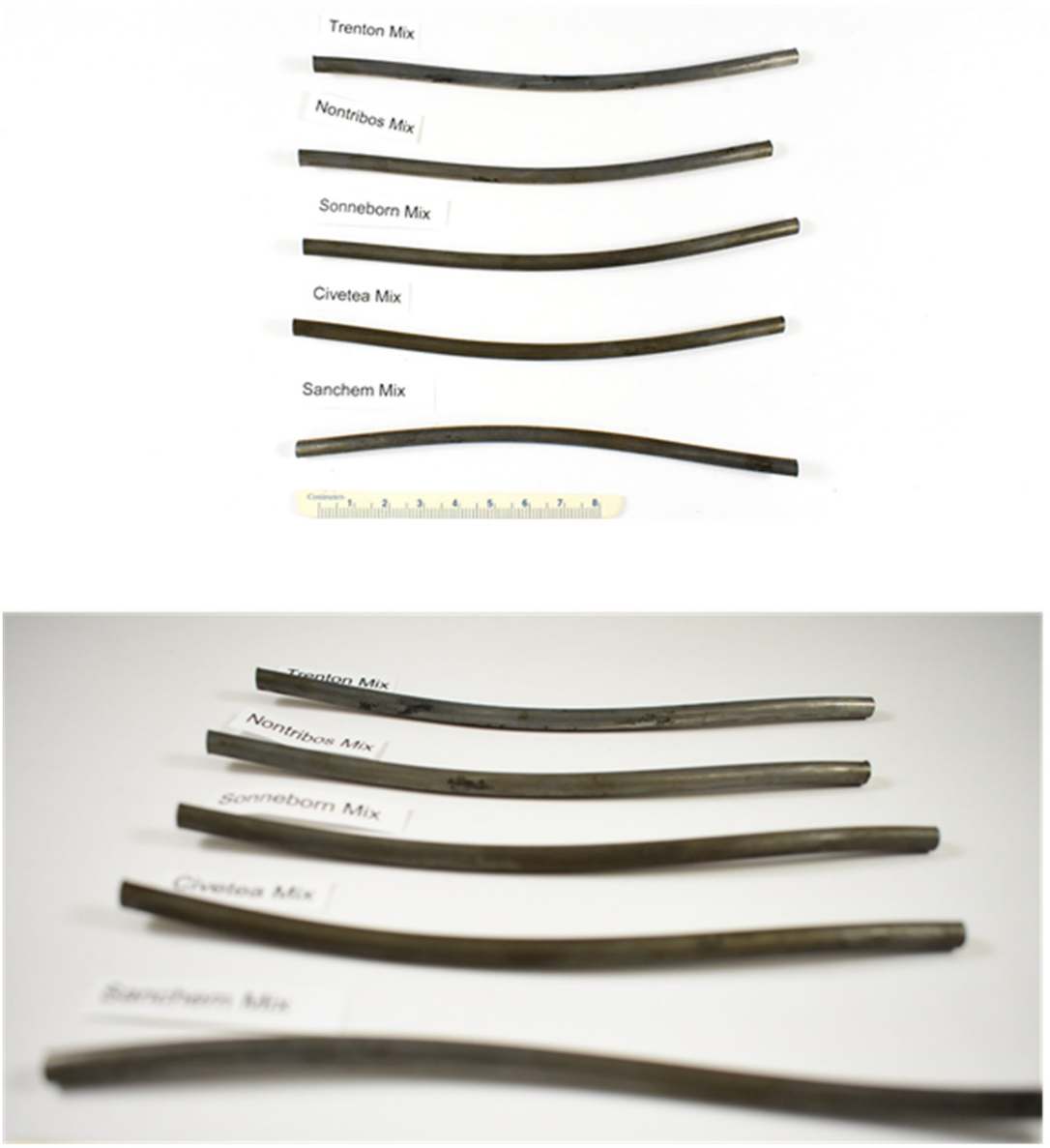


Figure 290. Coated wires sprayed with 0.6 milliliter of solution containing fungi mix

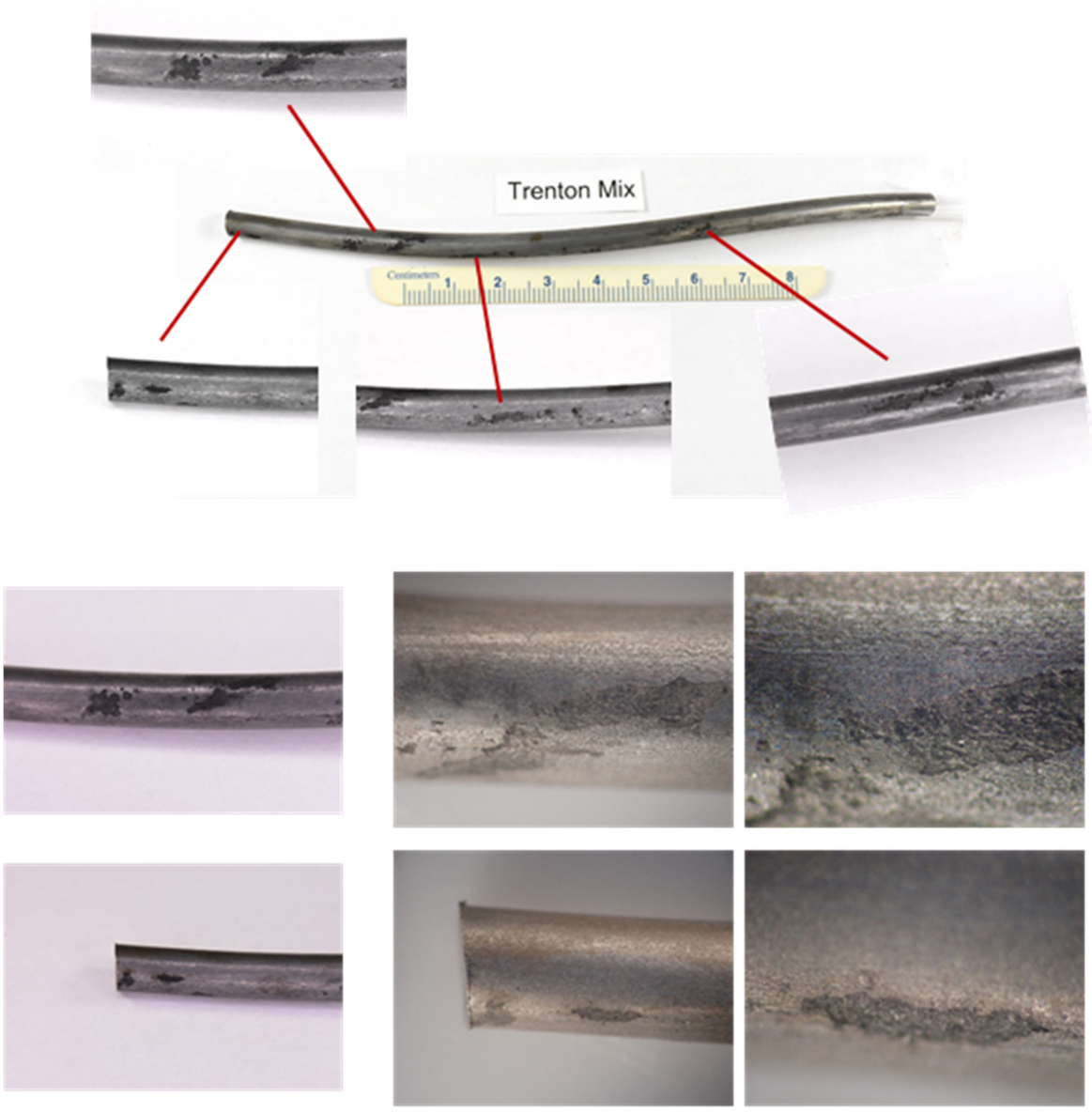


Figure 291. Coated with filler Trenton and sprayed with 0.6 milliliter of solution containing fungi mix (close-up 1).

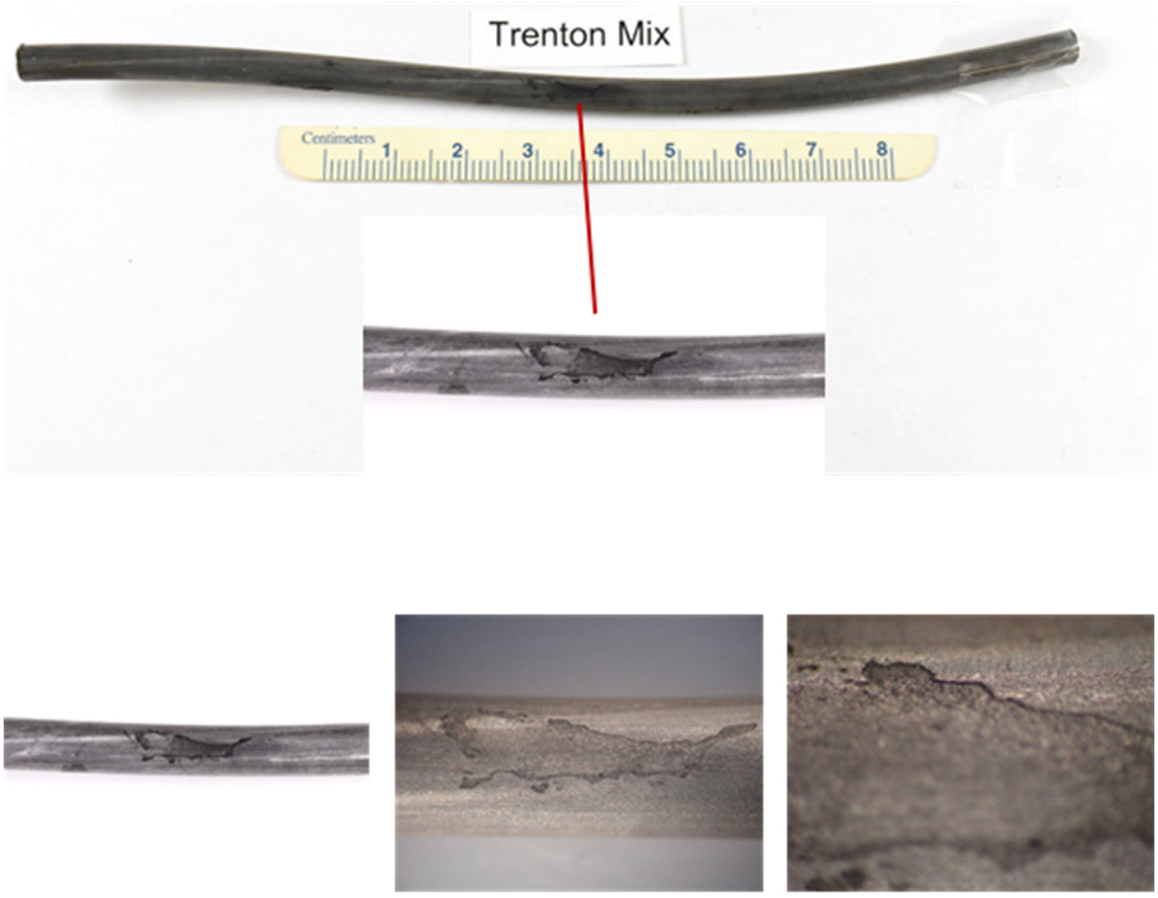


Figure 292. Coated with filler Trenton and sprayed with 0.6 milliliter of solution containing fungi mix (close-up 2).

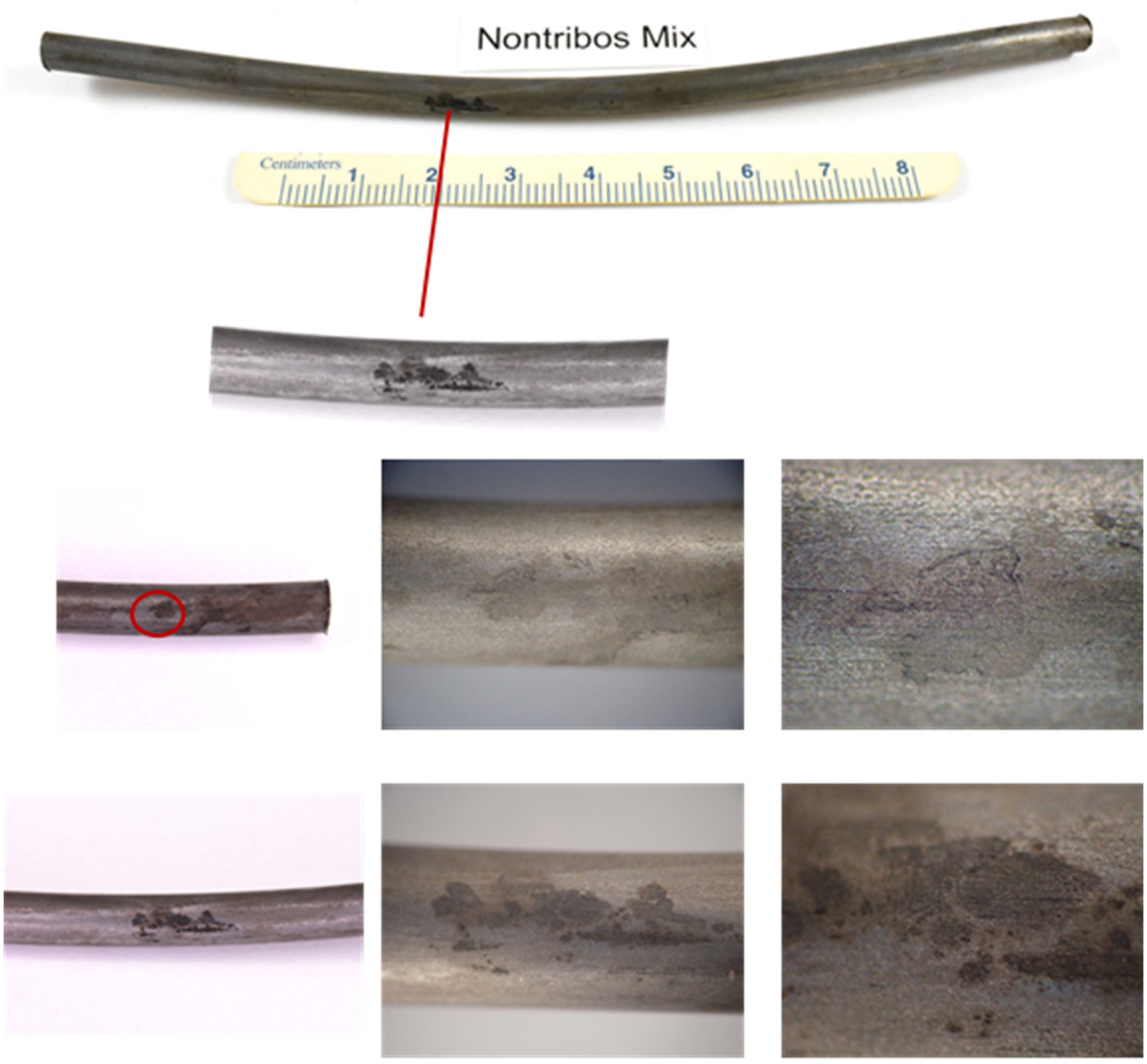


Figure 293. Coated with filler Nontribos and sprayed with 0.6 milliliter of solution containing fungi mix (close-up 1).

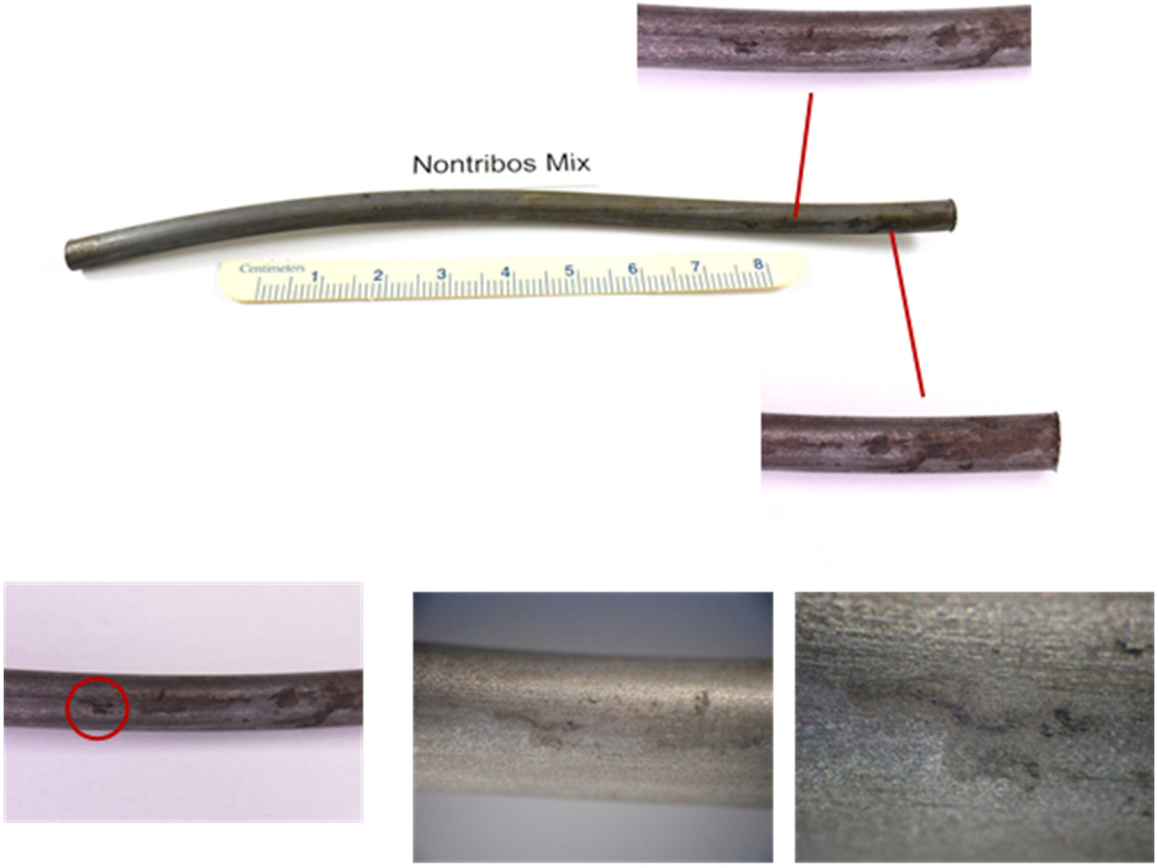


Figure 294. Coated with filler Nontribos and sprayed with 0.6 milliliter of solution containing fungi mix (close-up 2).

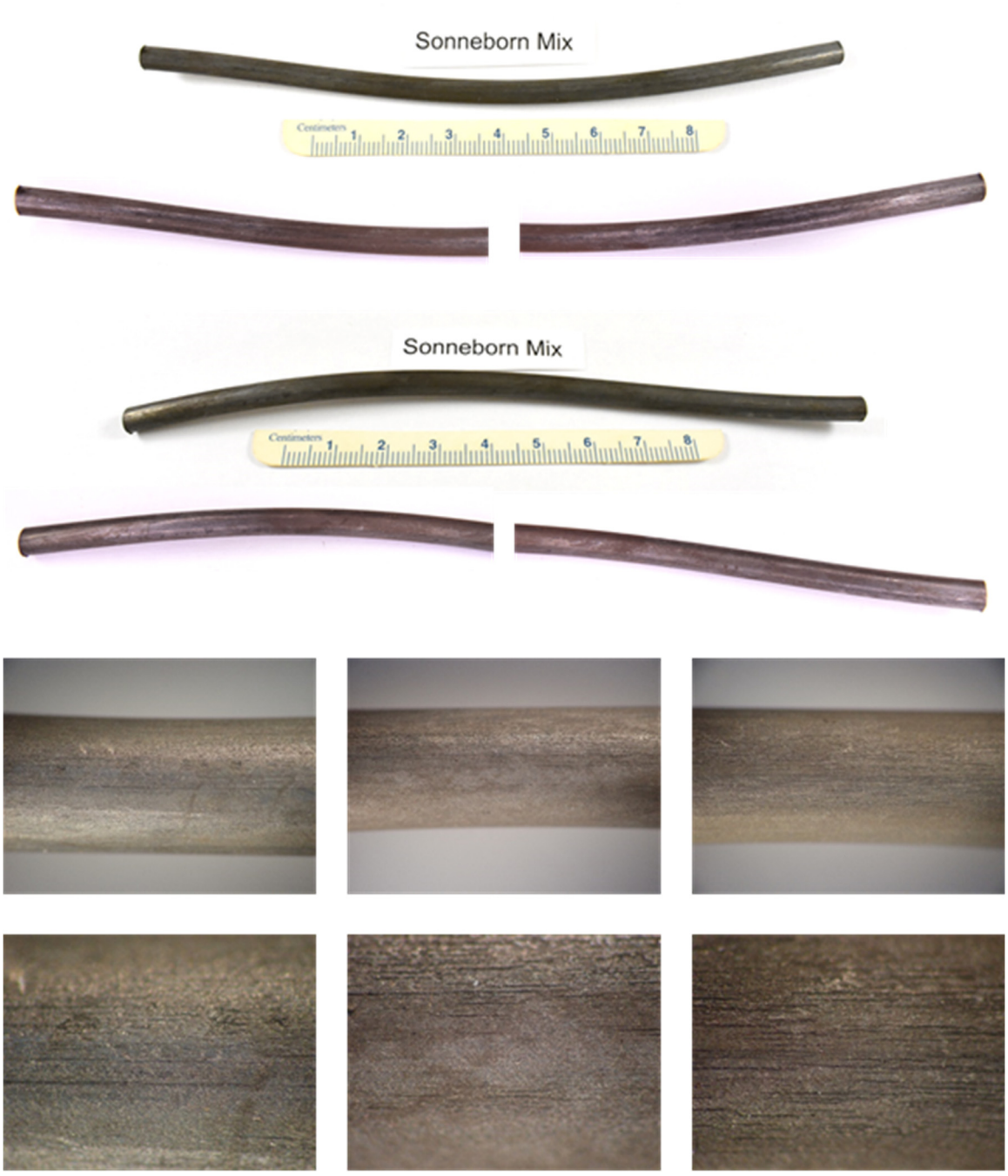


Figure 295. Coated with filler Sonneborn and sprayed with 0.6 milliliter of solution containing fungi mix (close-up 2).

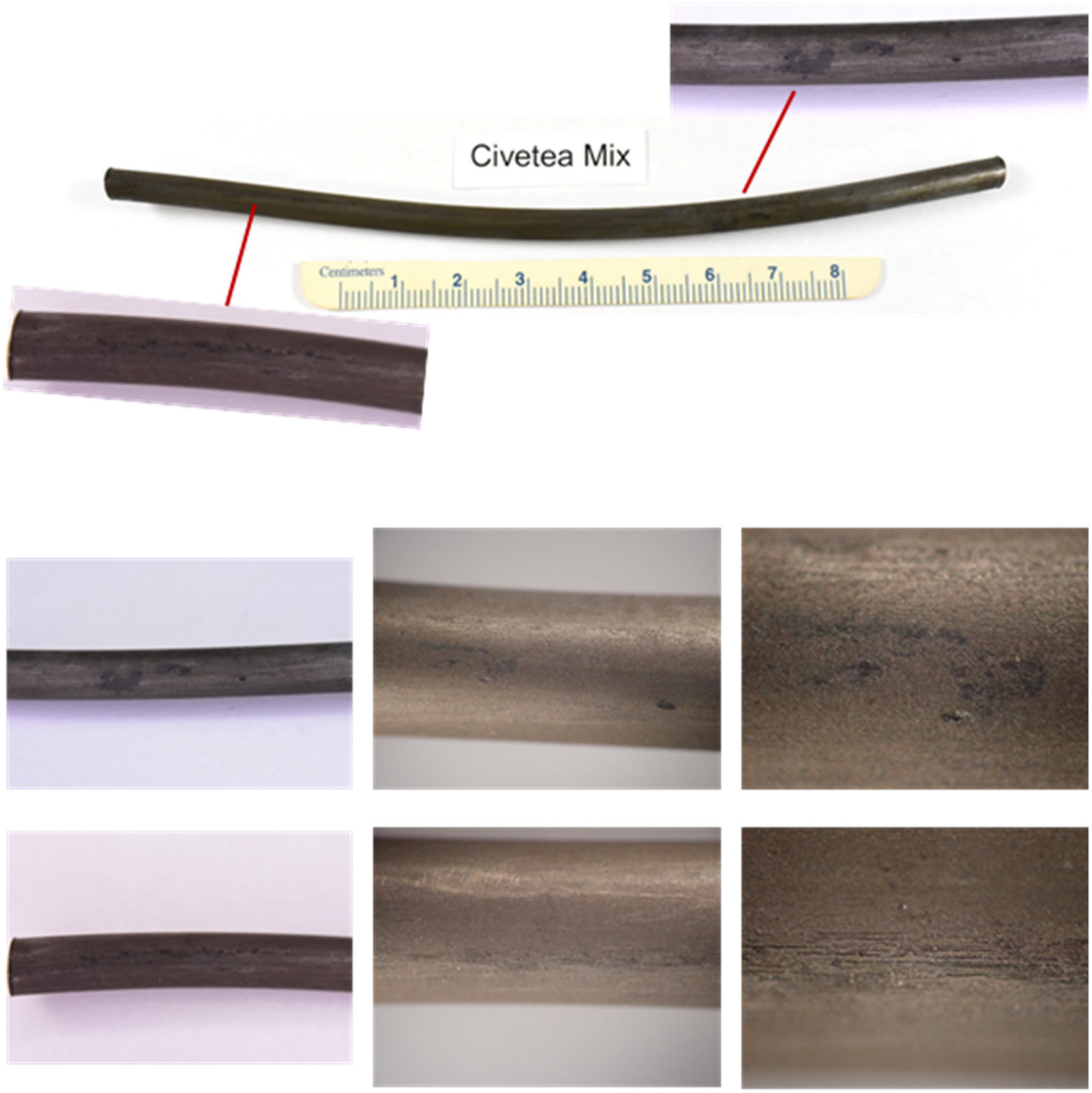


Figure 296. Coated with filler Civetea and sprayed with 0.6 milliliter of solution containing fungi mix (close-up 1).

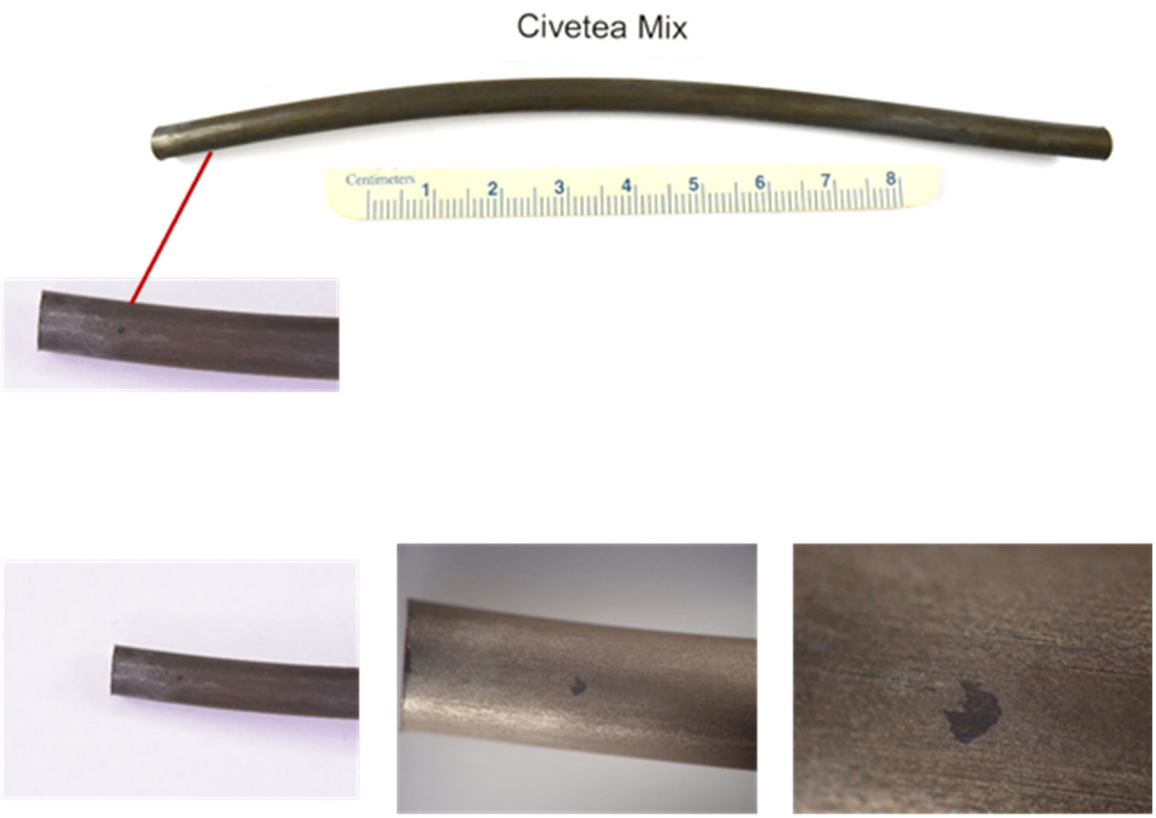


Figure 297. Coated with filler Civetea and sprayed with 0.6 milliliter of solution containing fungi mix (close-up 2).

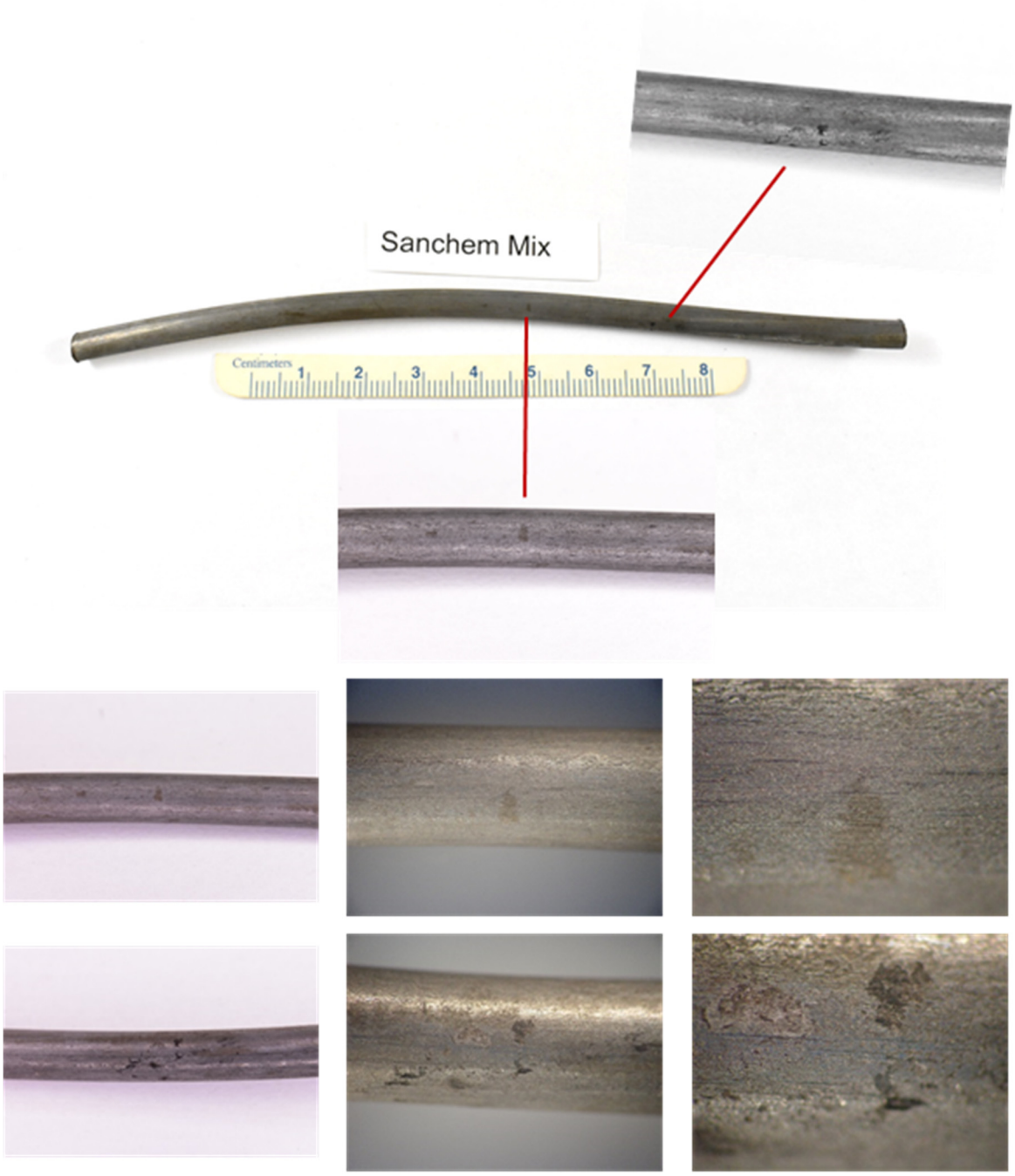


Figure 298. Coated with filler Sanchem and sprayed with 0.6 milliliter of solution containing fungi mix (close-up 1).

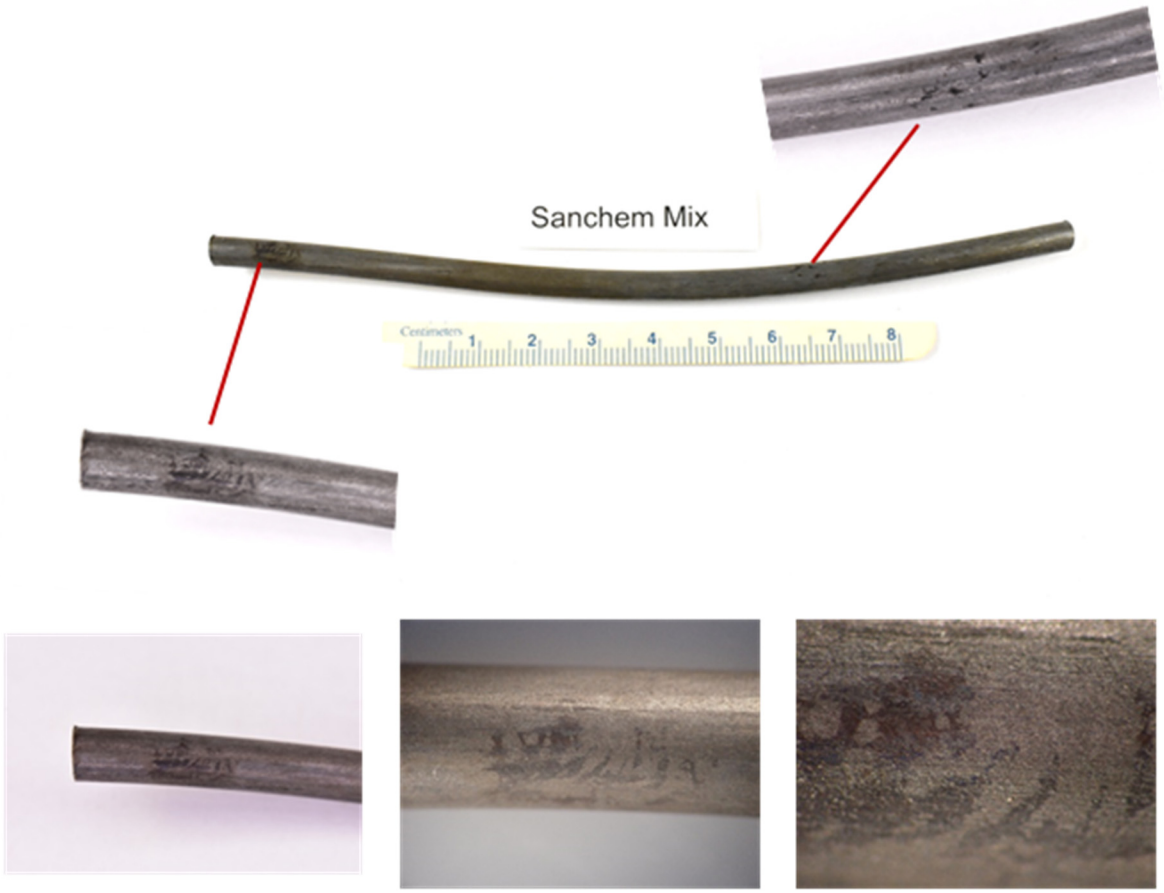


Figure 299. Coated with filler Sanchem and sprayed with 0.6 milliliter of solution containing fungi mix (close-up 2).

Figure 300. Coated with filler and sprayed with 0.6 milliliter of solution containing fungus PC (full view)

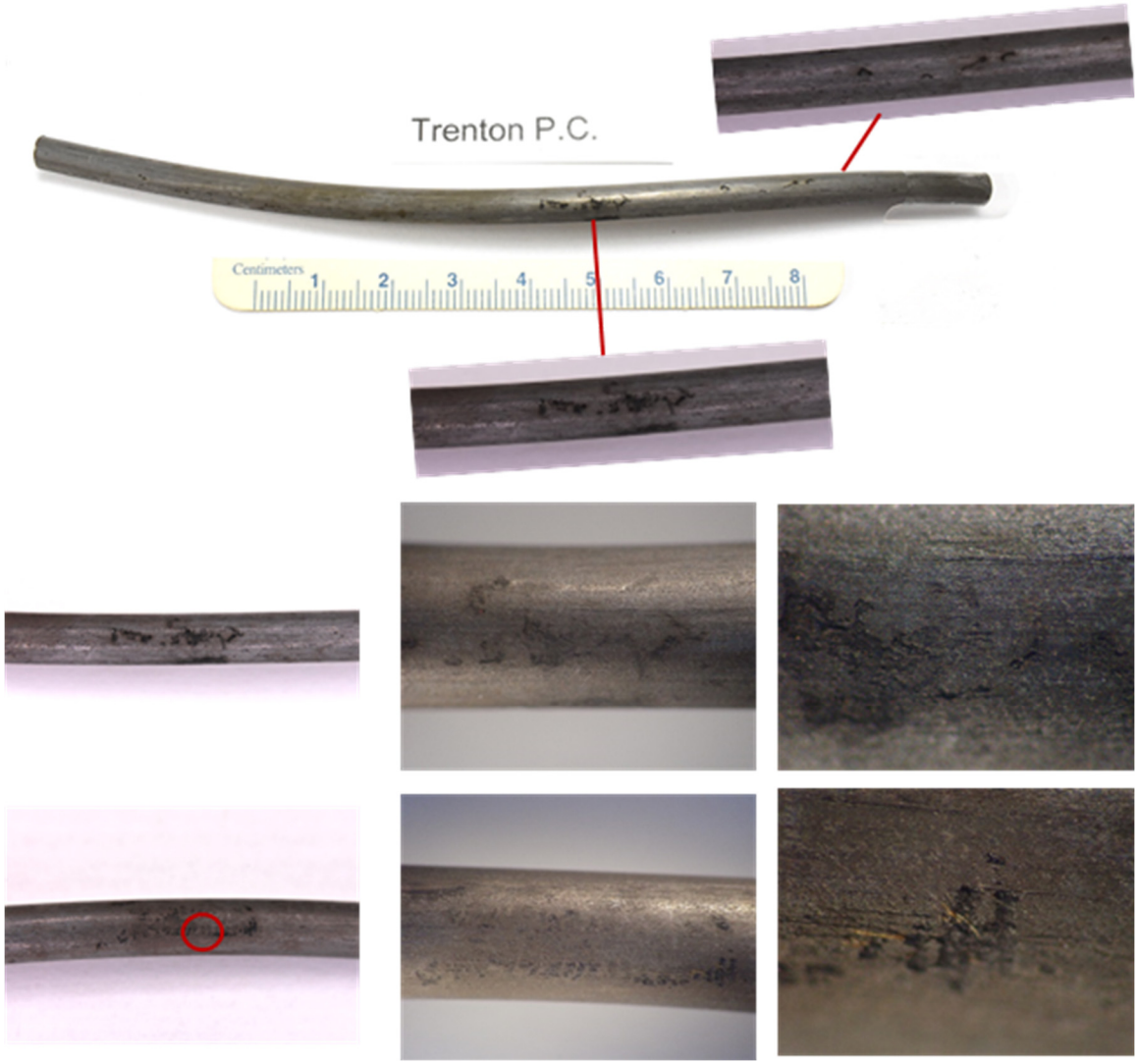


Figure 301. Coated with filler Trenton and sprayed with 0.6 milliliter of solution containing fungus PC (close-up 1).

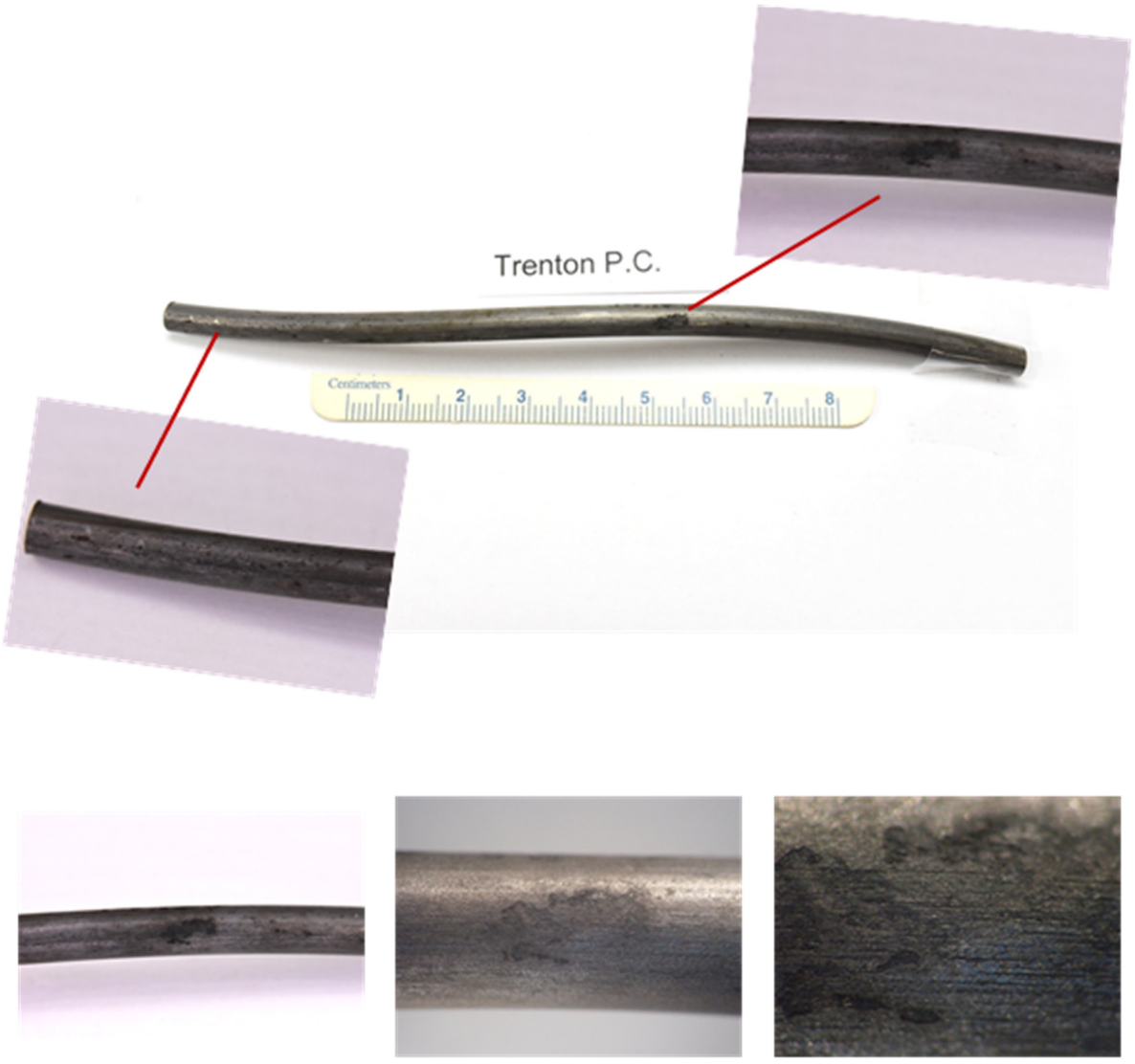


Figure 302. Coated with filler Trenton and sprayed with 0.6 milliliter of solution containing fungus PC (close-up 2).

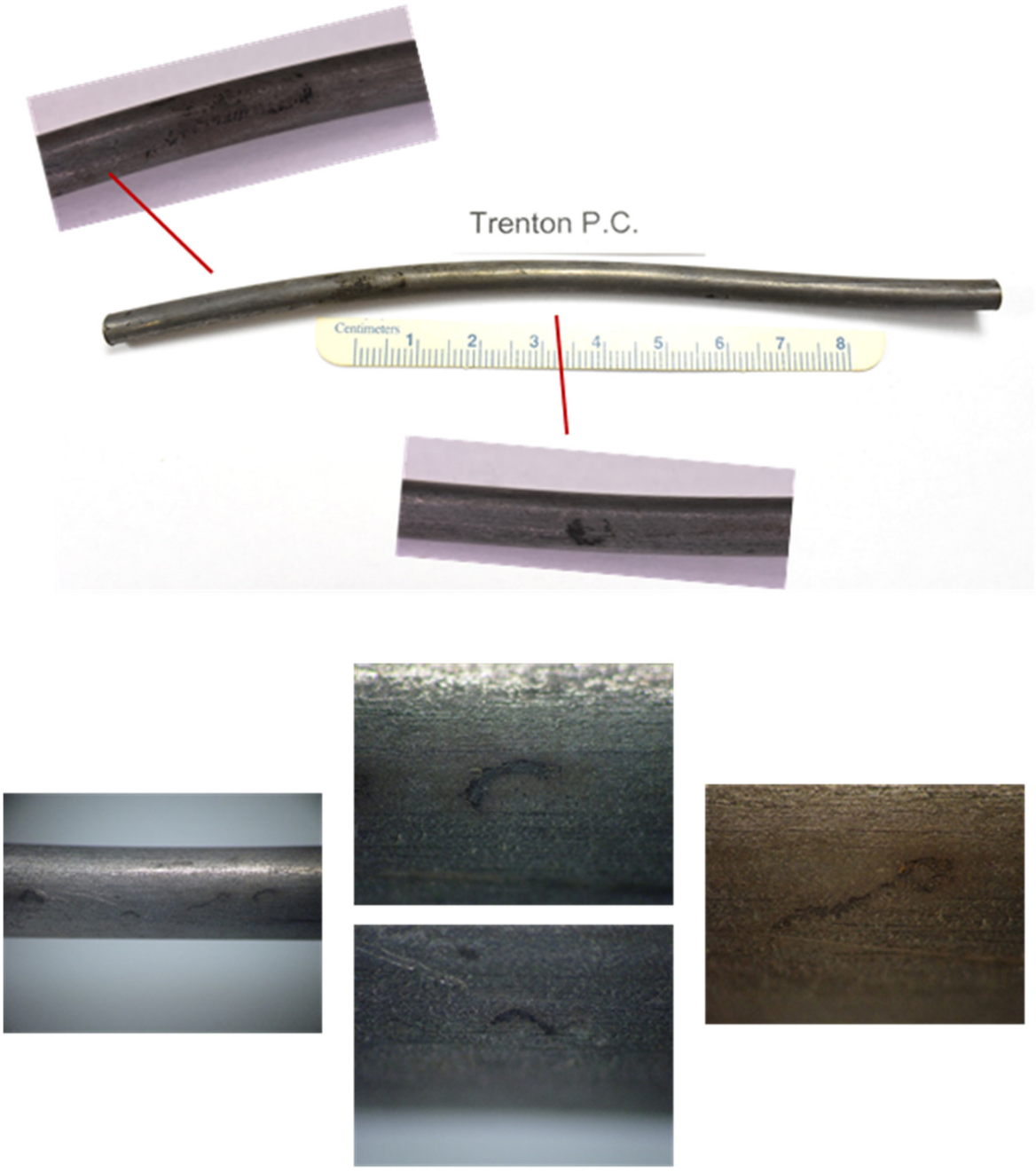


Figure 303. Coated with filler Trenton and sprayed with 0.6 milliliter of solution containing fungus PC (close-up 3).

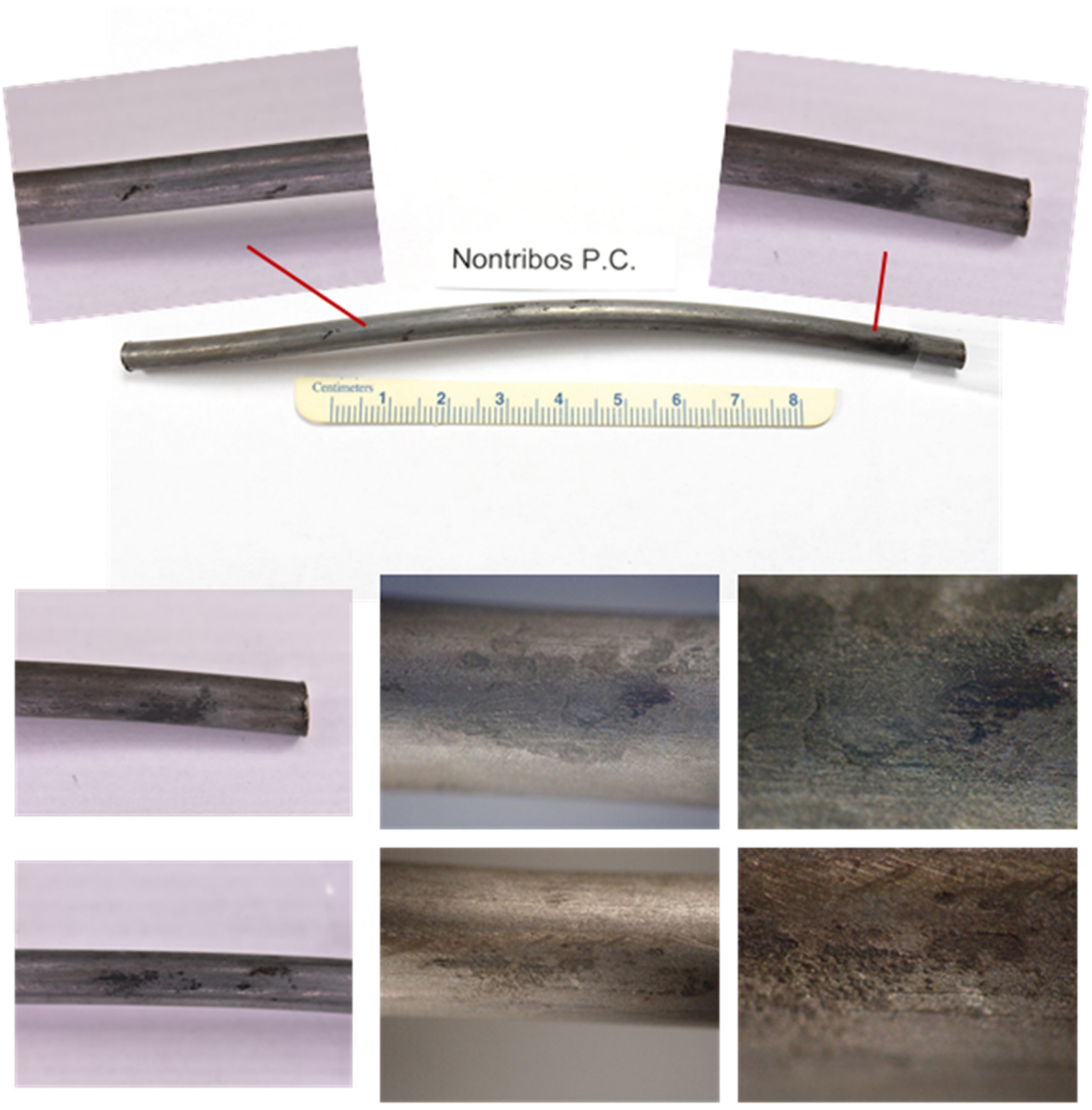


Figure 304. Coated with filler Nontribos and sprayed with 0.6 milliliter of solution containing fungus PC (close-up 1)

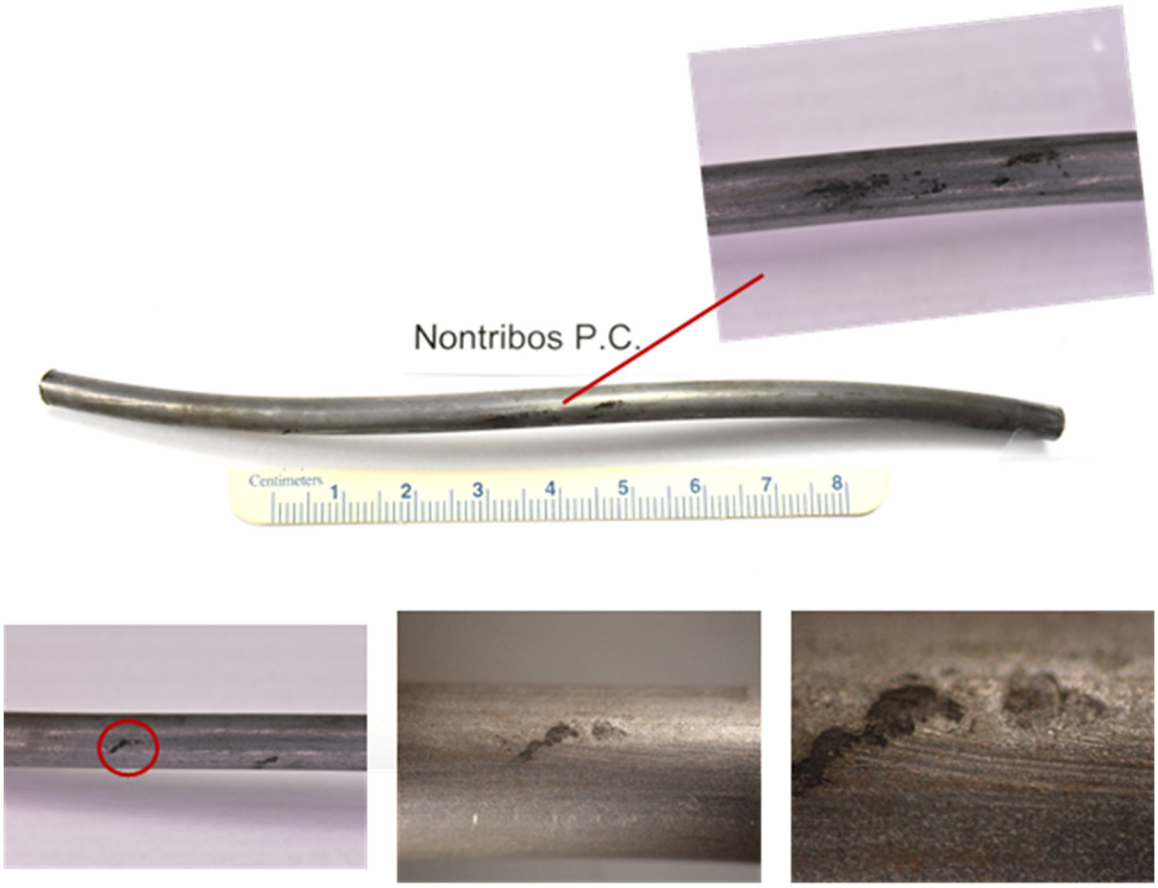
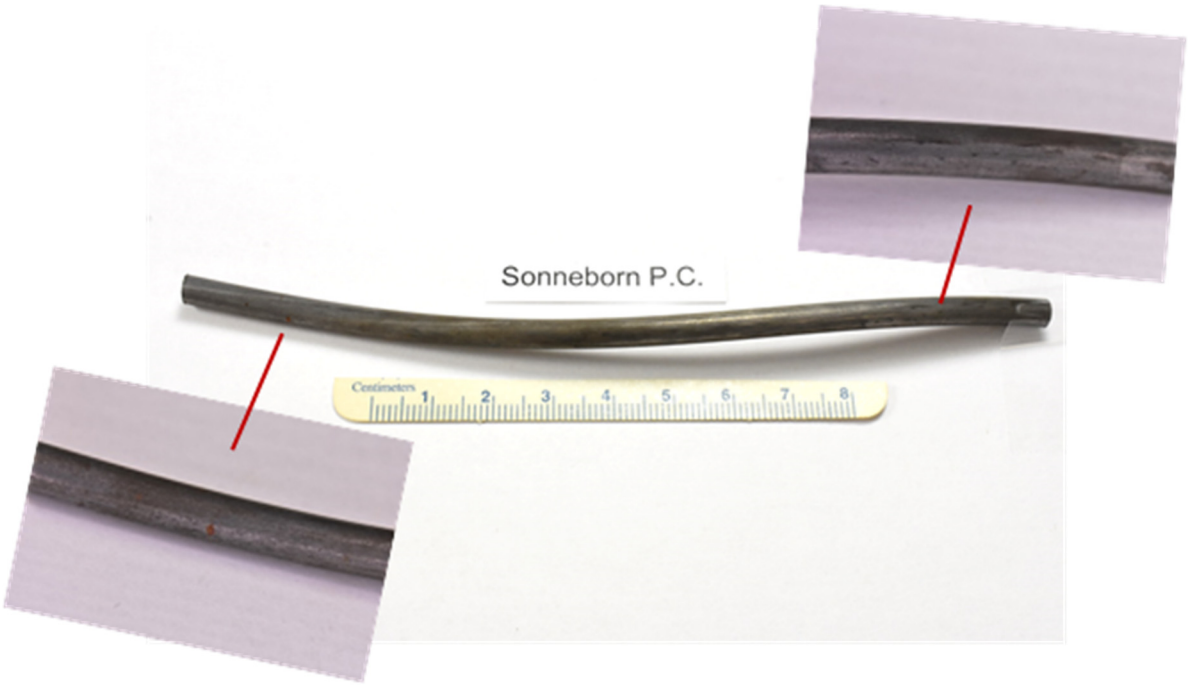


Figure 305. Coated with filler Nontribos and sprayed with 0.6 milliliter of solution containing fungus PC (close-up 2).



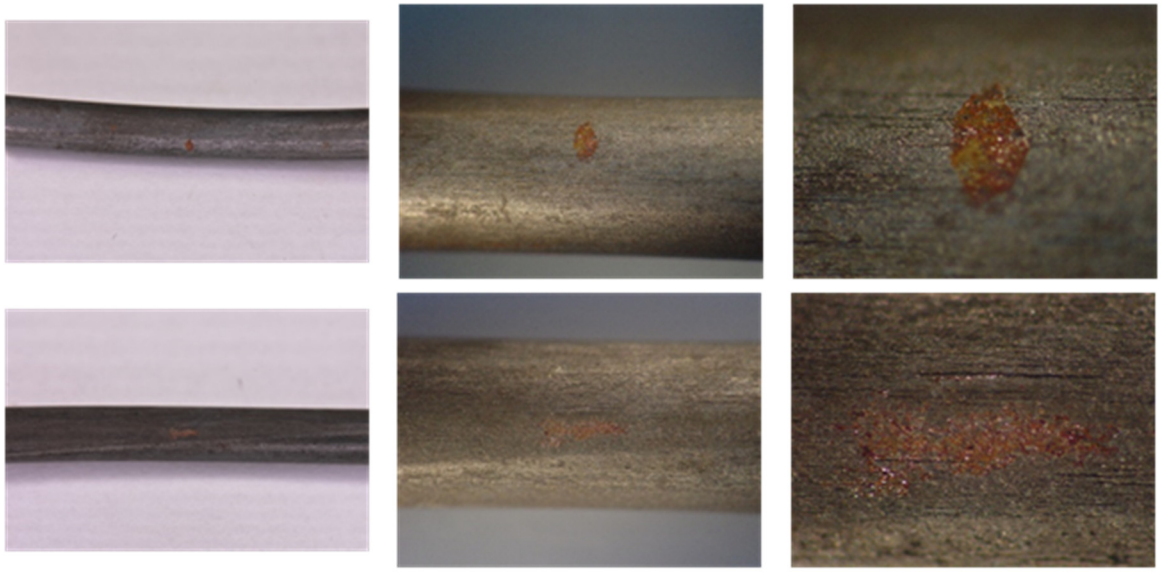


Figure 306. Coated with filler Civetea and sprayed with 0.6 milliliter of solution containing fungus PC (close-up 1).

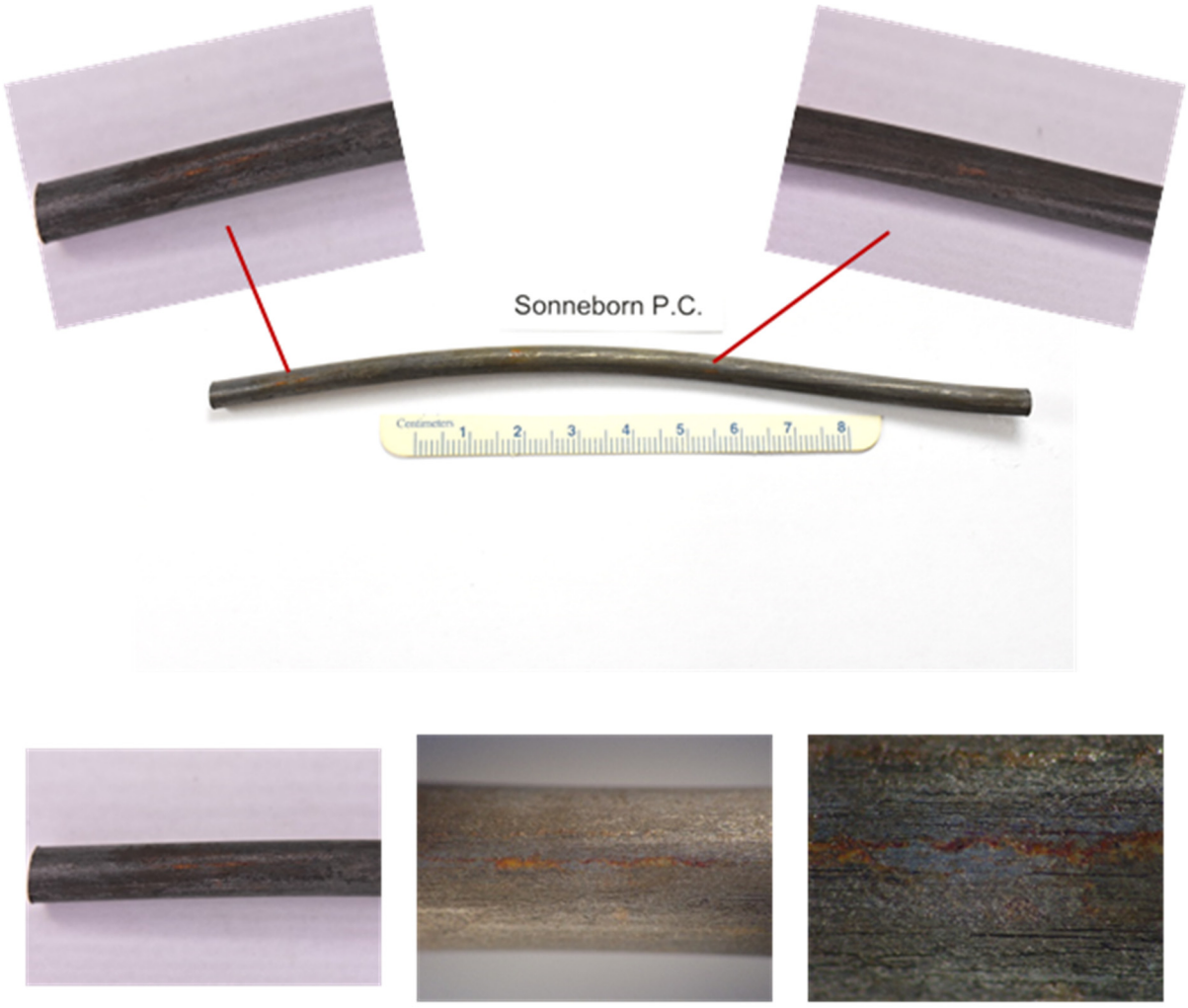


Figure 307. Coated with filler Civetea and sprayed with 0.6 milliliter of solution containing fungus PC (close-up 2).

Figure 308. Coated with filler Sanchem and sprayed with 0.6 milliliter of solution containing fungus PC (close-up 1).

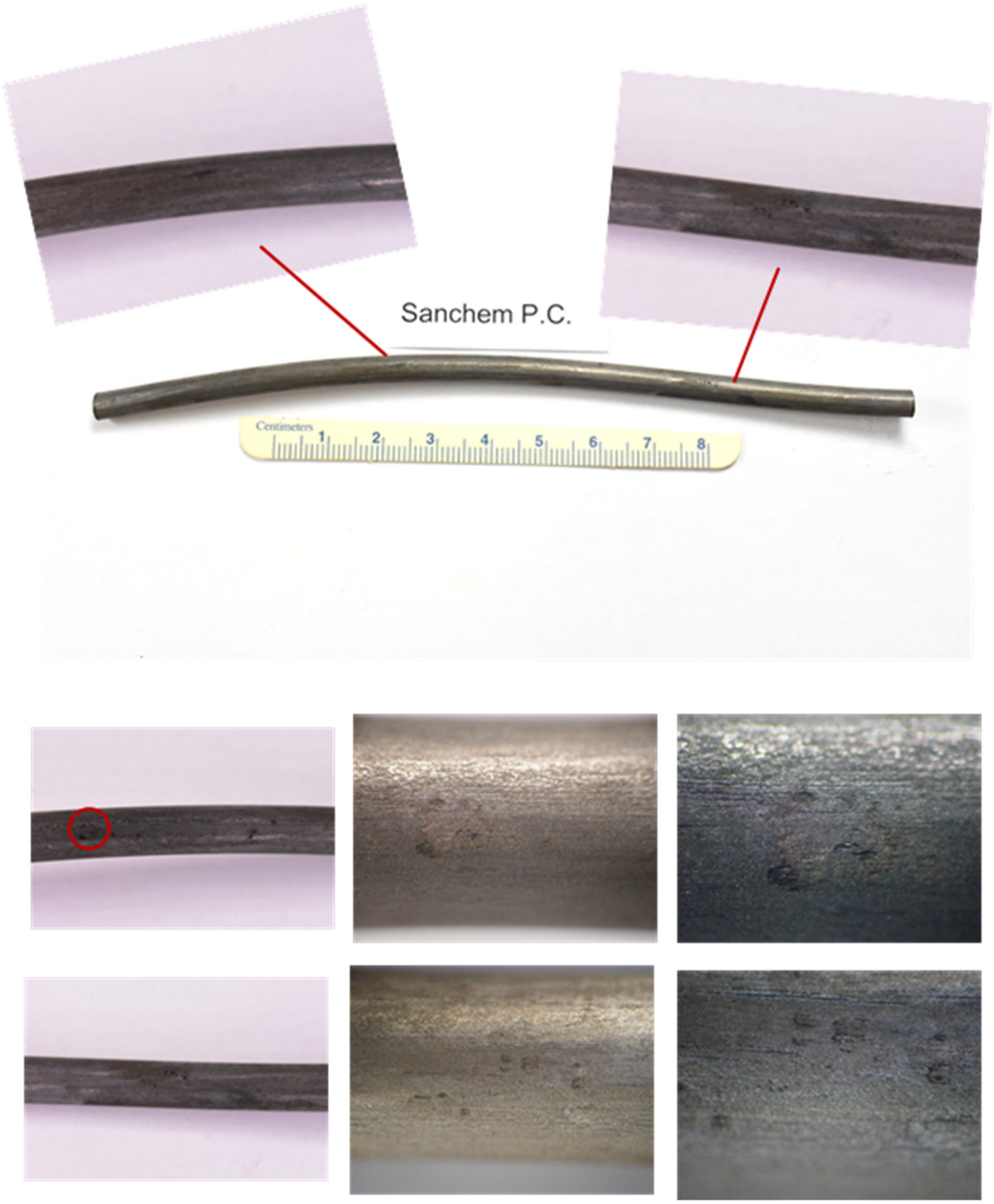


Figure 309. Coated with filler Sanchem and sprayed with 0.6 milliliter of solution containing fungus PC (close-up 2).