NILU TR : 17/84
REFERANSE: E-8339
DATO : DESEMBER 1984

AN ELECTROCHEMICAL TECHNIQUE FOR MEASUREMENTS OF TIME OF WETNESS

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ISBN-82-7247-544-8

FORORD

Denne rapporten inneholder publikasjonen til:

"Third international conference on the durability of building materials and components" i Helsingfors, august 1984.
Ved konferansen ble publikasjonene presentert som deler av hovedrapporter innen større områder. Publikasjonen ble omtalt av dr. Keith Martin innenfor "General Report 5: Environmental factors affecting service life: Effects, measurement and classification", under kapitlet "Evaluation of ageing factors". Utdrag av hans rapport vedlegges som appendix.

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AN ELECTROCHEMICAL TECHNIQUE FOR MEASUREMENT OF TIME OF WETNESS

Abstract

An electrochemical technique for measurement of time of wetness (TOW) is described. The method is based upon measurement of the current generated in electrolytic Cu/Cu cells at the time of atmospheric exposure. When this current exceeds a "wet" threshold value TOW is recorded by the instrument. The threshold value can be adjusted and calibrated to account for the humidity conditions occurring on the surface. The instrument which allows for recording of 6 sensors simultaneously is fully automatic with computerized data handling and analyses (NILU-WETCORR). The Cu/Cu sensor is now being further developed and optimized and some results are given.

1 INTRODUCTION

The rate of atmospheric corrosion of metals is a complicated function of a great number of factors such as the surface wetness, temperature, content of air pollutants, nature of the metal, and the amount, structure and composition of corrosion products on the metal surface.

To obtain a better understanding as to how these parameters affect atmospheric corrosion, a technique which makes it possible to establish the instantaneous value of the corrosion rate would be an important development. For the last ten years much effort has been devoted to modifying electrochemical techniques for determination of the atmospheric corrosion rate and/or the time of wetness (TOW) (1). Most of these methods make use of galvanic cells that generate potential/current across the electrodes when a humidity film occurs. TOW is then defined as the time when the potential or current exceeds pre-determined threshold levels. In the USA Sereda's method based on potential changes in Cu/Zn sensors is now proposed as an ASTM- standard for TOW-determination (2).

A different approach for simultaneously measuring of TOW and atmospheric corrosion rate is based on early work by Tomashov (3), who used a galvanic corrosion battery that consisted of alternate copper and iron plates and measured the galvanic current. Kucera and Mattson at the Swedish Corrosion Institute (SCI) (4) were the first to develop this technique for practical measurements, which also included the development of electrolytic cells and an electronic integrator for continuously recording TOW and cell current. This work was also taken up by NILU, and through a common Nordic research program SCI and NILU used the method in several studies during the seventies (5).

This paper first briefly summarizes the measurement principle and some of the TOW results obtained with the SCI/NILU method. Secondly, the limitations of this method are outlined and the further development of the method and some of the most recent obtained results are presented.

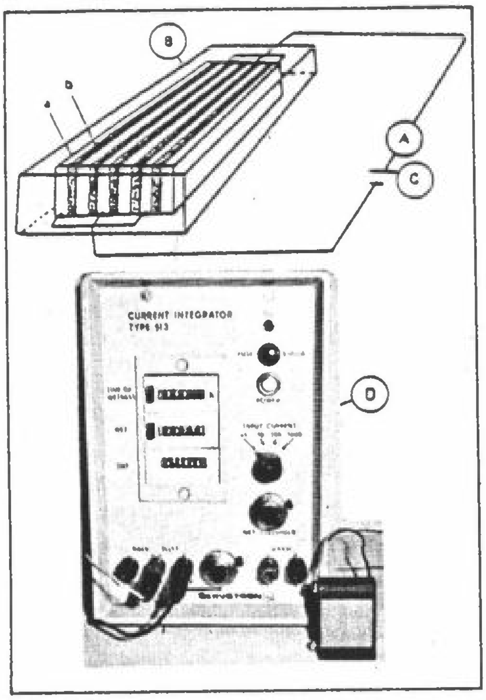
2 THE SCI/NILU METHOD

2.1 Measurement principle and equipment

The electrochemical equipment has been described earlier in detail (4). The SCI/NILU equipment consisted of an electrolytic cell, a zero resistance ammeter, and a built- in DC voltage source with which the impressed voltage could be varied within the range 0-2 volts (Fig. 1a). The cells consisted of a varying number of 0.5 or 1 mm thick plates insulated from one another with polycarbonate foil, the total electrode area being in all cases about 6.5 cm^2. When the cells were exposed to the atmosphere the current flow was continuously measured with the zero resistance ammeter. On exposure to the outdoor atmosphere the cell current varied over a wide range depending on the precipitation, humidity and pollution conditions, as shown in Fig. 1b.

From this cell current response the TOW was defined as the time when the cell current exceeded a fixed threshold value. In most studies this threshold value was set at 1 μA (= 1/6.5 $\mu A/cm^2$) (5).

To be able to use the cell current for quantitative purposes an electronic current integrator was developed which also recorded the TOW. Conventionally the integrated currents and TOW had to be read on the counters of the integrator at desired time intervals.



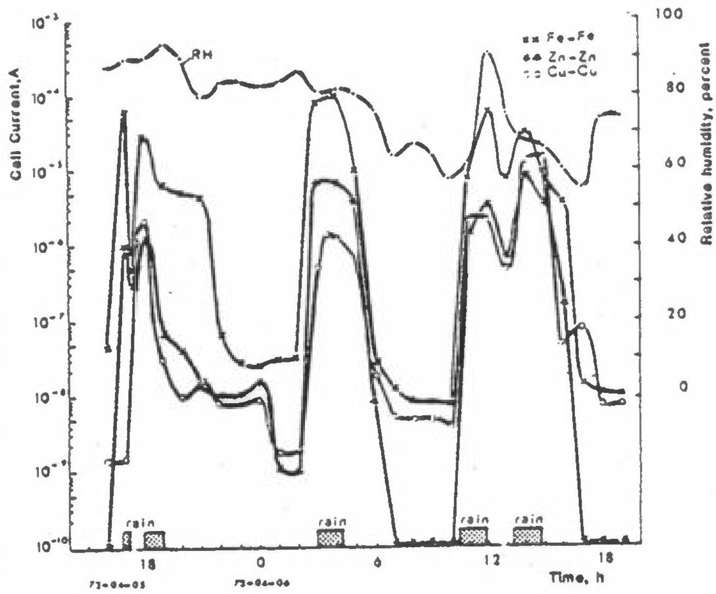


Fig. 1a. General arrangement of electrochemical device for measurement of atmospheric corrosion: A. zero resistance ammeter. B. electrochemical cell of electrolytic type. a) electrodes, b) insulators. C. external emf. D. electronic current integrator and TOW recorder.

Fig. 1b. Cell current in electrolytic cells of the iron-iron zinc-zinc, and copper-copper types, all with an imposed potential of 100 mV, and the relative humidity in Stockholm over the period of 5 April - 7 April 1973 (1).

2.2 Relation between TOW, cell current and humidity parameters

Most of the results obtained may in principle be represented by the measurements shown in Fig. 2. The TOW recordings were compared to the relative humidity continuously recorded by termohygrograph.

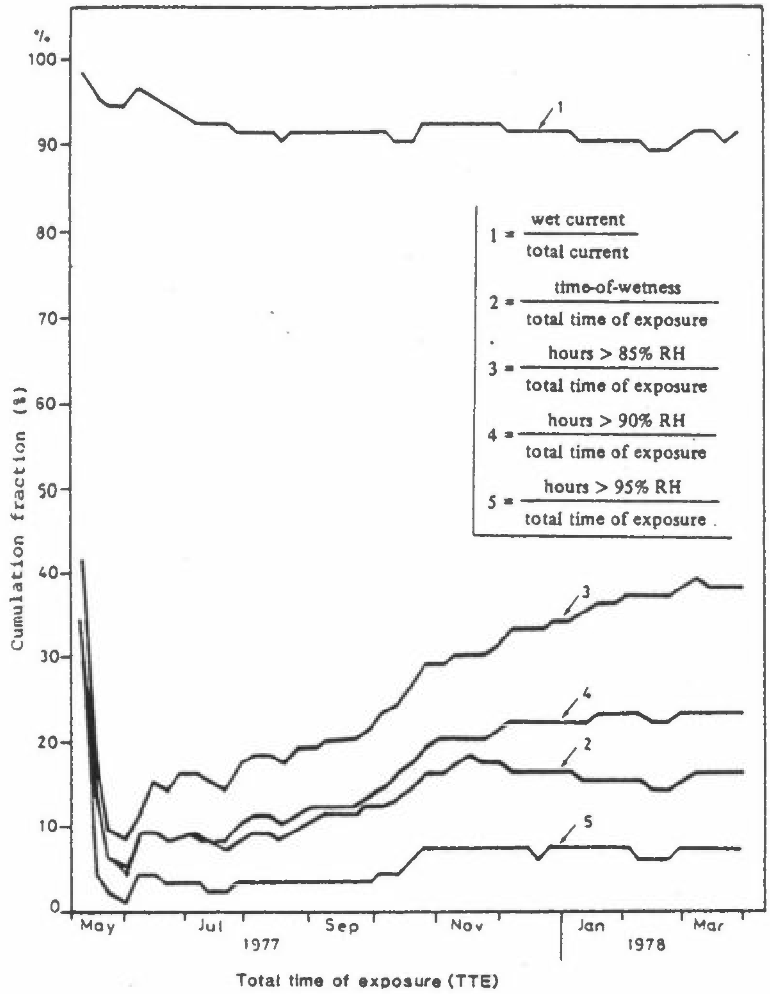


Fig. 2. Cumulative fraction of "wet" current, TOW and relative humidity in measurements performed at roof of NILU building in period 1977-05-11 to 1978-03-31 (5).

Fig. 2 shows that the fraction wet current/total current (curve 1) is between 90-95% all the time, and that the TOW (curve 2) is almost identical to the time of relative humidity RH > 90% (curve 4) for the first 7 months of exposure. This corresponds well with experience showing that the atmospheric corrosion rate of carbon steel is closely correlated with the time of RH > 80-90%. In a proposed standard for classification of the atmospheric corrosivity, the TOW, calculated from termohygrographic data in ambient air is defined as the time when relative humidity exceeds 80% with the temperature simultaneously being above

0^0C (6). The deviation between the TOW (curve 2) and the RH > 90% (curve 4) during the winter exposure from Nov-77, may be explained by the fact that the cumulative fraction calculated has not taken into account that the temperature should simultaneously be above 0^0C.

Although the wet current thresholds should be somewhat adjusted, the nature of the results obtained, as illustrated in Fig. 2, were considered sufficiently promising to justify further development of the method.

2.3 Limitation of the method

From the experience gained in the 1970's the two following items should be developed further:

1. Full automatization of measuring equipment and computerized data handling/analysis.

2. Smaller cells that are less expensive and more easily produced.

3 THE NILU-WETCORR METHOD

3.1 Computerized current integrator and TOW recorder (NILU-WETCORR)

The development of automatic and computerized instrumentation began in 1980, initiated by the need for an automatic multichannel compact unit for the study of the corrosivity on different parts of the car body (7).

The block diagram for the computerized instrument is shown in Fig. 3.

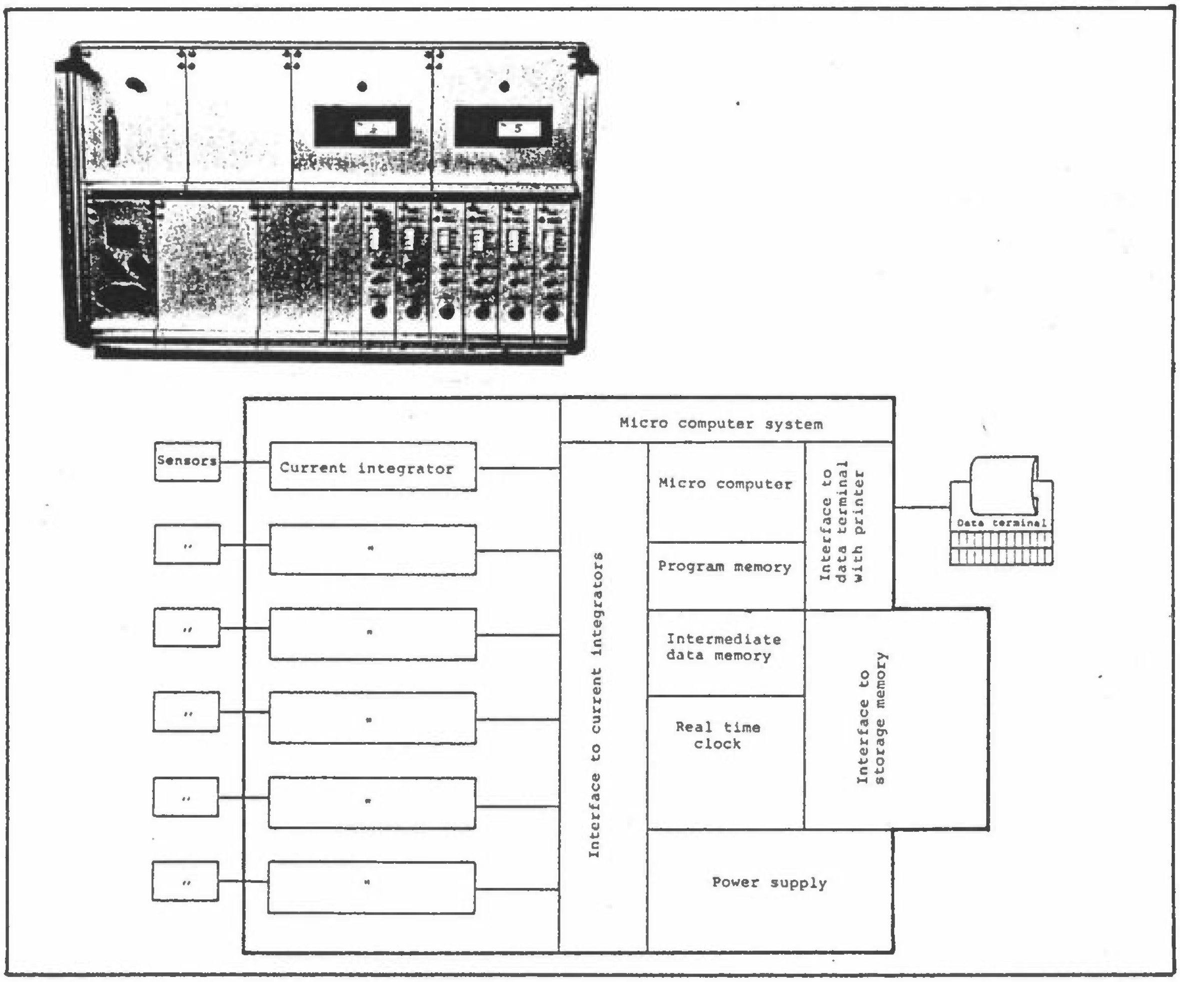


Fig. 3. Picture and block-diagram of the NILU-WETCORR.

The instrument has the following properties:

- 6 independent voltage sources and current integrators for which the impressed potential and wet threshold level could be separately chosen.
- The integrated current is recorded on either a "dry" channel, or on a "wet" channel when the wet current threshold is exceeded. The TOW is then simultaneously recorded on another channel. The integrating time interval can be chosen in either minutes (5, 10, 15, 20, 30) for "wet" current recording or in hours (1, 2, 3, 4, 6, 8, 12, 24)

for "dry" current recording. The time interval changed automatically from hours to minutes when the current in one cell exceeds the wet threshold value.

- The integrator can record the accumulated current for the entire exposure period or for each separate sampling interval.
- The time for each measurement is labeled with month, day, hour and minute. The impressed potential can be either DC or AC at requested frequency. The instrument can be programmed from a data terminal.
- All data in the storage memory can be read directly into our central NORD 100 computer.

After testing a twelve channel prototype in the car study in 1981, the instrument was rebuilt in our laboratory in 1982/83. It is named NILU-WETCORR (time of WETness and COrrosion Rate Recorder) after its main functions.

In the car study we were able to map the time of wetness on 12 different points of the car body as a function of driving and weather conditions. The electrochemical cells used were the old Fe/Fe type earlier described (2.1).

3.2 The electrochemical sensor

The alternative to the original type of cell seems to be cells produced by the well developed thin film technique from the electronic industry, i.e. the same type of cells as used by Sereda (2). We applied our measuring principle to some of Sereda's cells in a study of TOW on partly isolated high voltage equipment in climate chamber testing (8) and the results were promising (see 3.3).

TOW and corrosion rate is a specific variable for each metal. In principle the sensor should therefore consist of the metal in question. We have found producers who can supply us with cells of Cu/Cu, Fe/Fe, Zn/Zn and Al/Al. The price of each cell is about 10 dollars.

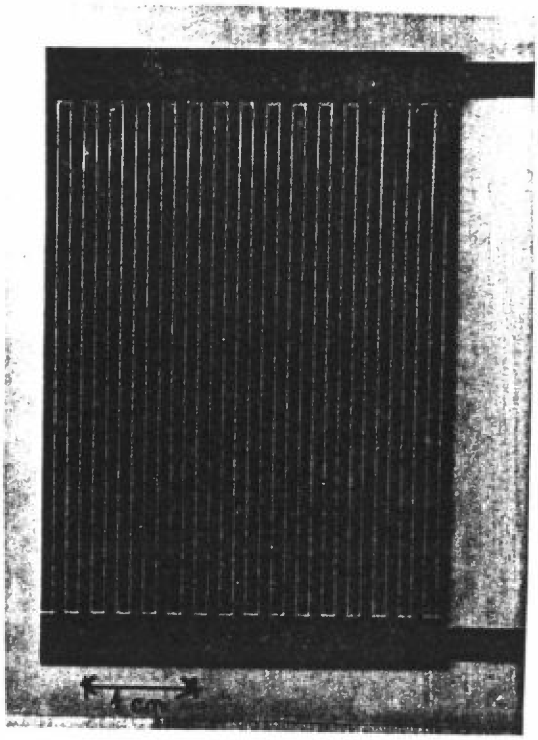


Fig. 4. Cu/Cu thin film sensor.

For the purpose of having a TOW sensor that can be used to measure TOW in or on the surface of other materials we have decided to use the Cu/Cu sensor. A prototype of the cell is shown in Fig. 4. At present we are calculating the potential/-current distribution in the cell with the aim of calculating the optimum cell dimensions. Decreasing the total length of the "fingers" decreases the current output, while decreasing the distance between the fingers increeases the the current output. However, smaller distances also increases the risk of a short circuit. Some experiments conducted in the process of developing TOW sensors follow.

4 EXPERIMENTS

4.1 TOW by climate chamber testing of partly isolated high voltage equipment

On partly isolated high voltage equipment built in compact boxes problems are often encountered with flash-over on switches due to humidity and condensation. In order to investigate this problem a climate chamber study was conducted by which the NILU-WETCORR method was used to record the microclimatic humidity and condensation conditions, i.e. on the surface of the various parts of the equipment. Sereda cells were used as sensors (Fig. 5a).

Fig. 5b shows the placement of cells on the equipment in the climate chamber. The sensors are attached to the equipment with two-sided tape. Cells 1 to 4 are the Au/Zn type, while cell 5, which is in the same place as cell 4, is a Au/Cu cell. Imposed voltages to the cells were 100 mV, and wet current threshold was set to 0.2 μA.

Fig. 5c shows hourly mean values of the cell current for the five cells during the three day test. The course of the temperature and RH (calculated) inside the chamber is shown in the upper part of the same figure. The test program for the chamber consisted of "wet" periods, where the temperature and RH were increased from about 10^0C/ca 70% RH to 35-40^{0}C/ 90-100% RH, and "dry" periods where temperature and RH were rapidly decreased to the first level.

All four Au/Zn sensors are sensitive to humidity formation on the surface, and there are significant and systematic differencies in the time course of

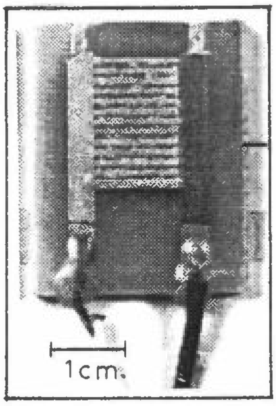


Fig. 5a. Au/Zn sensor type Sereda.

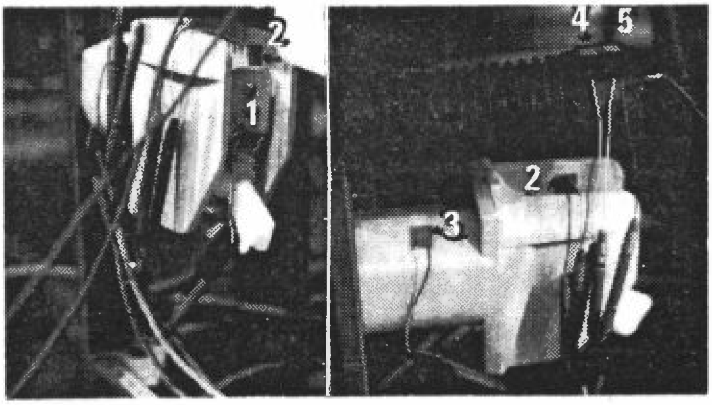


Fig. 5b. Placement of TOW sensors on high voltage equipment by climate chamber testing.

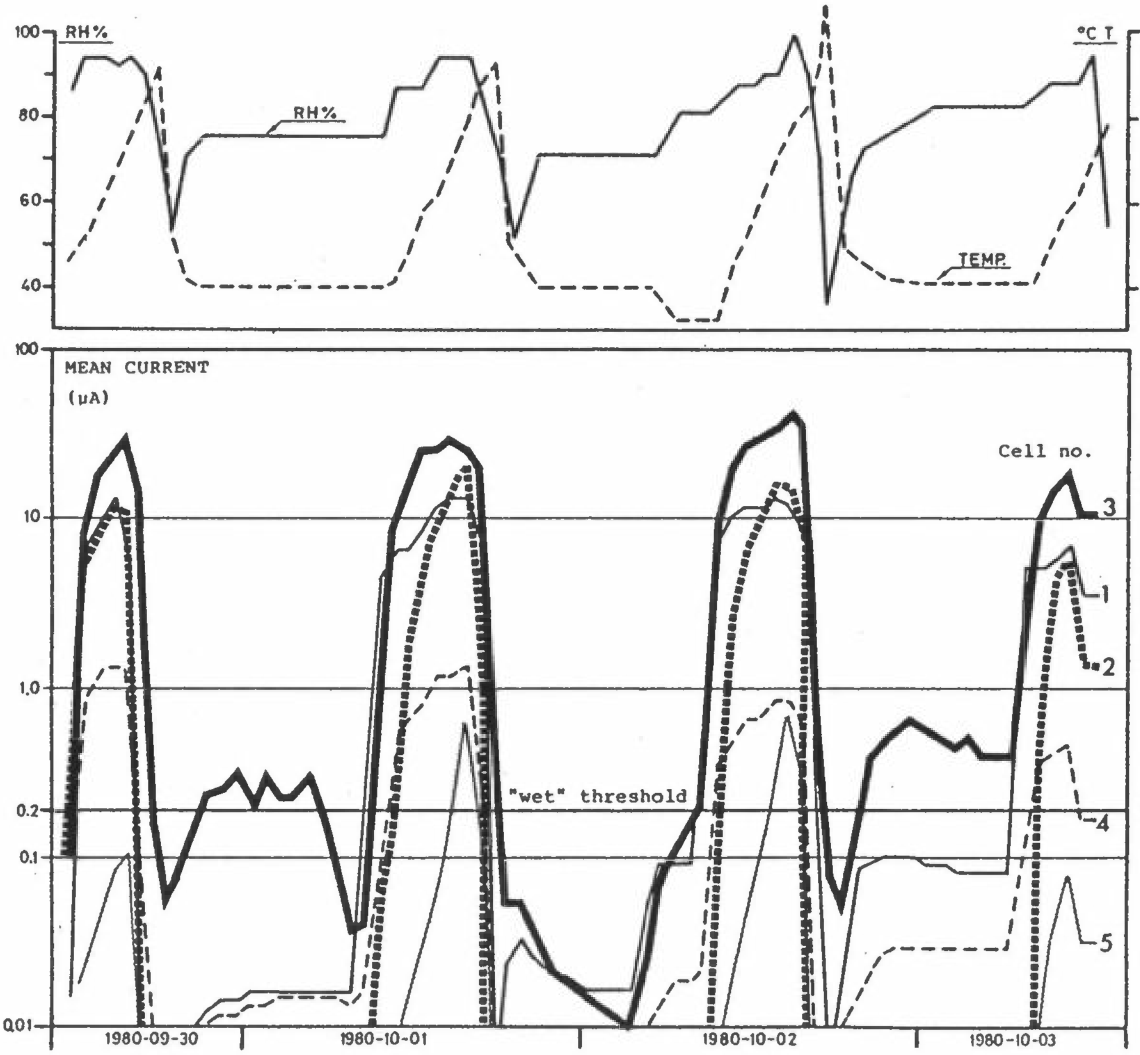


Fig.5c. Temperature - RH characteristics of chamber and current response in TOW sensors.

cell currents on the various points. It also seemed that an increase in temperature increased the cell current more than did an increase in RH alone. In all wet periods the current was highest in cell 3 and lowest in cell 4, the mean current were 20-30 μA and about 1 μA, respectively. Cell 1 and 2 had about the same mean current in the wet periods, i.e. about 10 μA.

The end values for TOW in the five cells were:

Table 2: TOW as recorded by NILU-WETCORR method in climate chamber testing.

Cell no.	TOW (h)	%
1	23.30	32
2	17.30	24
3	45.34	62
4	20.67	28
5	2.71	4

Qualitatively these results may be explained reasonably well in relation to the mass and heat capacity of the underlying surface at each measuring point. A more detailed discussion is given in (8), but the following conclusions can be drawn:

1. The cells display clear and significant differences in current response for "wet" and "dry" periods.
2. The cells also show significant differences in current response for different measuring points on the surfaces. Quantitatively these differences may be reasonably explained in terms of mass and heat capacity etc. of the underlying surface at the different measuring points.
3. The results show that the method may be used for study of humidity conditions on various surfaces.

4.2 Cu/Cu cells - reproducibility, cell dimensions and A.C. polarization

In order to study the reproduciblity and the effect of cell dimensions and A.C. polarization commercially made Cu/Cu cells were exposed in field and climate chamber tests.

4.2.1 Field test

Six identical Cu/Cu cells (area 15.4 cm^2 - Fig. 4) were exposed on the roof of the NILU building for six days. Impressed potential to the cells was 100 mV D.C. and the wet threshold was 0.2 µA. TOW recorded was compared to ambient air temperature and RH, as measured by a TH-graph.

The results are summarized in Table 2. More detailed results are given in (9). The cells show a high level of reproducibility and reliability, five of them recording exactly the same % TOW while the sixth cell is about 3% lower. The accumulated amount of cell current varies within $\pm$ 5%.

Table 2: % TOW as measured by NILU-WETCORR with 6 Cu/Cu cells and as calculated from TH-graph data at NILU for the period 1982-09-15--21.

Cell no.	TOW %	Amount current (A.s)	% TOW calculated from TH-data as: $\frac{\text{time of RH > x \% and T > } 0^0\text{C}}{\text{Total exposure time}} \cdot 100\%$		
			x=80%	x=85%	x=90%
1	41	1.33			
2	41	1.47			
3	38	1.38			
4	41	1.46	62	52.5	34
5	41	1.43			
6	40.5	1.44			
Mean	40.5	1.42			

In principle the wet threshold level should be chosen in such a way that TOW is recorded when humidity on the surface is high enough to cause a significant increase in the corrosion rate. The recorded TOW approximately equals the TOW calculated from TH-graphic data as the time above 85-90 RH.

4.2.2 Climate chamber test

Eight Cu/Cu cells, six of them with an area of 15.4 cm^2 and two with an area of 1.5 cm^2, were tested in a climate chamber for approximately three months. The various changes made during th test and the results are shown in Table 3. Fig. 6 shows the current output from the 6 cells during the period 1984-01-26--31.

From the field and climate testing of Cu/Cu TOW sensors the following conclusions can be drawn:

1. The reproducibility and reliability of the cells are very good. The lower % TOW for the first test period in the climate chamber is due to somewhat lower humidity during the first three days of exposure.

2. The ratio of the cell current output for the two cell sizes, i.e. 7.5, are almost equal to the ratio between the total length of the electrode fingers in the two cells, i.e. 6.2.

3. A.C. compared to D.C. polarization of the cell increases the current output by a factor of about 5-6, according to comparison of cell 2 + 4 with 5+6 in the third test period.

Table 3: Time of wetness and integrated cell current in Cu/Cu cells in climate chamber testing at NILU 1983-12-20--1984-01-31. Cells <u>and</u> measuring principle are changed during experiment. Climate program: 8h 20^{0}C/70%RH and 8h 40^{0}C/98%RH.

Period 1: 1983-12-20-1984-01-06. Reproducibility.

Cell no.	Dimension* (mm)	Area (cm^2)	Pot. (mV)	"Wet" t.h. (μA)	% TOW	"Wet" Current (A.sec.)	"Dry" Current (A.sec.)
1	32x48x1(0.2)	15.4	100	0.2	39.1	0.33	0.064
2	"	"	"	"	30.7	0.24	0.070
3	"	"	"	"	30.2	0.23	0.064
4	"	"	"	"	37.0	0.30	0.061
5	"	"	"	"	33.8	0.26	0.075
6	"	"	"	"	36.4	0.29	0.065
Mean					34.5±4.5	0.28	0.067

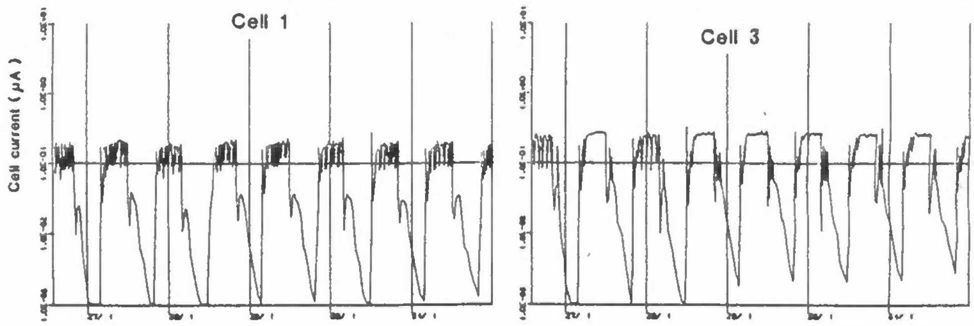
Period 2: 1984-01-12--01-17 - Reproducibility and effect of cell area (cell no. 1 and 3).

Cell no.	Dimension* (mm)	Area (cm^2)	Pot (mV)	"Wet" t.h. (μA)	% TOW		"Wet" Current (A.sec.)		"Dry" Current (A.sec.)	
1	10x15x1(0.2)	1.5	100	0.2	0		0		0.015	
2	32x48x1(0.2)	15.4	"	"		46.9		0.096		0.031
3	10x15x1(0.2)	1.5	"	"	0		0		0.023	
4	32x48x1(0.2)	15.4	"	"		64.7		0.150		0.025
5	"	"	"	"		50.7		0.110		0.032
6	"	"	"	"		47.5		0.100		0.032
Mean					0	52.5	0	0.114	0.019	0.030

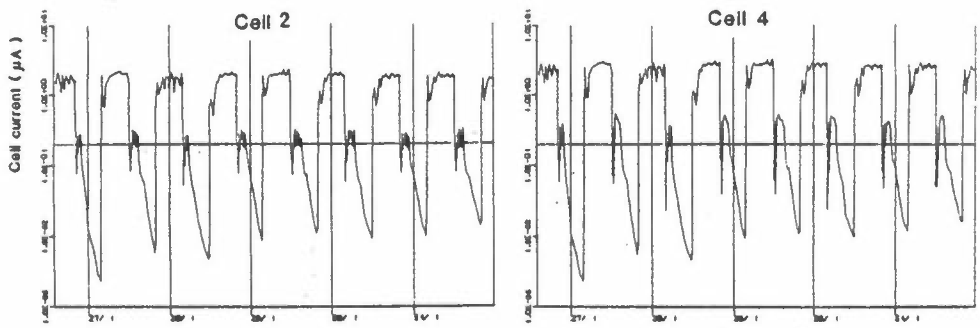
Period 3: 1984-01-19--01-31 - Reproducibility and effect of alternating polarization and "wet" threshold.

Cell no.	Dimension* (mm)	Area (cm^2)	Pot. (mV)	"Wet" t.h. (μA)	% TOW			"Wet" Current (A.sec.)			"Dry" Current (A.sec.)		
1	10x15x1(0.2)	1.5	100/30s	0.1	42.2			0.077			0.017		
2	32x48x1(0.2)	15.4	"	0.2		77.0			0.98			0.011	
3	10x15x1(0.2)	1.5	"	0.1	49.8			0.110			0.018		
4	32x48x1(0.2)	15.4	"	0.2		79.6			1.40			0.007	
5	"	"	100	0.2			47.4			0.19			0.038
6	"	"	"	0.2			44.1			0.15			0.036
Mean					46	78.3	45.8	0.094	1.19	0.17	0.018	0.009	0.037

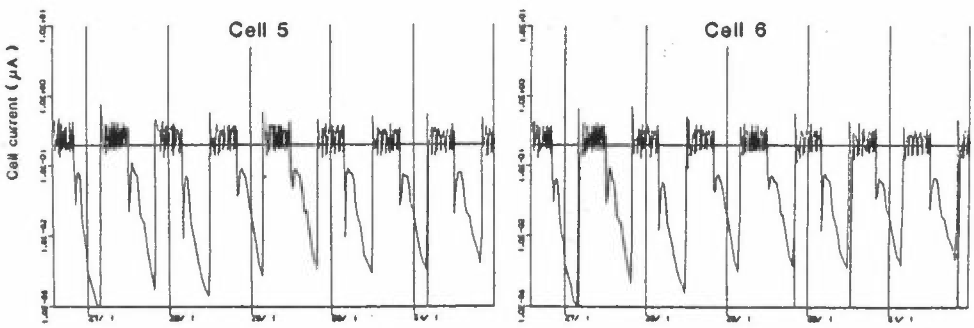
*Dimension = number of fingers x length of fingers x width of fingers (distance between).



a) Cell 1 and 3: Area 1,5 cm^2, ± 100 mV/30 sec, "wet" threshold 0,1 μA



b) Cell 2 and 4: Area 15,4 cm^2, ± 100 mV/30 sec, "wet" threshold 0,2 μA



c) Cell 5 and 6: Area 15,4 cm^2, ±100 mV D.C., "wet" threshold 0,2 μA

Fig. 6. Current response in Cu/Cu TOW sensors in climate chamber testing at NILU 1984-01-26--31 (Part of Period 3, Table 3).

4. The "wet" threshold can be adjusted to account for the real humidity conditions occurring on the surface of the cell, i.e. compare cell 1 + 3 in test period 2 with the same cells in test period 3

5. The peak values of the "wet" and "dry" currents differ by about two orders of magnitude.

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APPENDIX

Utdrag fra General Report 5 under
Third international conference on the durability
of building materials and components

VTT Symposium 51

Third international conference on the durability of building materials and components

Troisième colloque international sur la durabilité des matériaux et des éléments de construction

Volume 4

VALTION TEKNILLINEN TUTKIMUSKESKUS
STATENS TEKNISKA FORSKNINGSCENTRAL
TECHNICAL RESEARCH CENTRE OF FINLAND
ESPOO 1984

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GENERAL REPORT 5
ENVIRONMENTAL FACTORS AFFECTING SERVICE LIFE:
EFFECTS, MEASUREMENT AND CLASSIFICATION

Abstract

As a framework for reviewing this subject area, three environments are considered relating to either production, function, or ageing of building materials and components. It is proposed that these environments relate to initial material properties, failure criteria, and rate of deterioration which, linked together, provide a simple durability model. While the model implies that the most useful approach is a quantitative one, it requires a qualitative knowledge of how the environmental factors affect the property values in order to show which particular material property is important and what form the parameters should take to describe the environmental factors.

The papers presented provide additional qualitative information on concrete and timber and the performance of several specific composites, solar collectors and insulated walls; but they mostly relate to the function environment, and very few data are presented on rates of deterioration of materials in different ageing environments. Climate is discussed as an ageing environment with the main attention being given to global classification, and to opinions on levels of aggressivity for planning, but the meteorological data considered are often not the appropriate ageing parameters. The papers reveal a common interest in understanding the role of moisture, especially under cyclical conditions in cold climates, and several potentially

useful means for measuring moisture parameters are presented. A lack of information about the role of high temperatures and an appreciation of the limitations of using air temperature as an ageing parameter are also apparent. Future action to develop an understanding of these matters is suggested.

Keywords: Building materials, Durability, Models, Ageing environment, Climatic factors, Moisture parameters, Temperature parameters.

1 INTRODUCTION

The service life of a building component is the time from its installation to the time of its unserviceability because of some critical material property falling below a pre-defined level. This simple durability model is illustrated in Figure 1 where the change of the critical property with time has three important features: the initial value, the rate of decay, and the final unacceptable value. These material property values relate to the processes of production, ageing, and failure of the component, and each of these processes is dependent on the environment in which it occurs. Most of the work on the environment and service life concerns only the ageing process, with little recognition of the importance of the effects of the production environment and the environment associated with the functioning of the component and the failure criterion.

Typical examples of the influence of the production environment on the initial material property value are the effect of moisture curing conditions on the strength of concrete, of the sintering temperature on the flexural strength of ceramics, and of the extrusion temperature on the fracture toughness of plastics. While it is generally advantageous to durability (but not necessarily to economics) to adopt production conditions to maximise the initial material property value, this approach may also produce high rates of decay and an overall reduction in

4 EVALUATION OF AGEING FACTORS

Evaluation of ageing factors for particular localities may require either calculation from meteorological information or direct measurement. An approach that is being adopted for a number of factors is to make direct measurements and correlate them to some function of available meteorological data, so that site evaluations can be extended to other regions. The best known example is time-of-wetness.

4.1 Time-of-wetness (TOW) measurement

Although expressing some doubt about whether the same critical value of relative humidity applied to the atmospheric corrosion of all metals, Sereda (16) proposed that TOW be taken as the cumulative time for which the relative humidity exceeded 85% for studies of metal corrosion. This was based on measurements of surface wetness according to the potential developed across a Cu/Zn cell. Haagenrud, Henriksen, Danielsen and Rode /5.02/ report an alternative approach to measurement of TOW based on the current developed when moisture bridges Cu/Cu cells. They also report the development of computerized data handling from 6 automatic sensors operating simultaneously. In principle, one advantage is that similar cells are available in a range of other metals (Fe, Zn, Al) and so the cell could be made of the same metal as that being corroded. It would be of interest to determine whether or not this is significant empirically, because the measurement depends on other factors such as the selection of the threshold current characterizing wetness, which in turn depends on the geometry of the cell. Data presented for condensation on high-voltage equipment in a controlled-climate chamber are very interesting in showing how condensation may vary over the surface of a single component depending on factors, such as the mass, which influence heat capacity. The sensors were sensitive enough to discriminate between the different sites on the component, and so should be capable of aiding the design of components to reduce time-of-wetness and, consequently, corrosion. Very good repeatability and reproducibility were reported, and researchers can look forward to further results of application of the sensors to specific deterioration problems of building components. The early findings indicate that matters of detail such as degree of exposure and design of component will greatly influence time-of-wetness; and that the calculation of this parameter from meteorological data will only be applicable to limited building components, and even for fully exposed surfaces will need to be adjusted for factors such as orientation, slope, and shading by other components.

Similar developments with TOW are no doubt proceeding in a number of countries, and a discussion of these would be of interest. In Australia, we have developed a Cu/Zn sensor but are planning to use it to measure films of moisture on the actual surface being weathered (Figure 2). Trials are proceeding with representative surfaces of concrete and painted substrates, which slowly revolve with the sensor contacting them intermittently to register the emf of the cell.

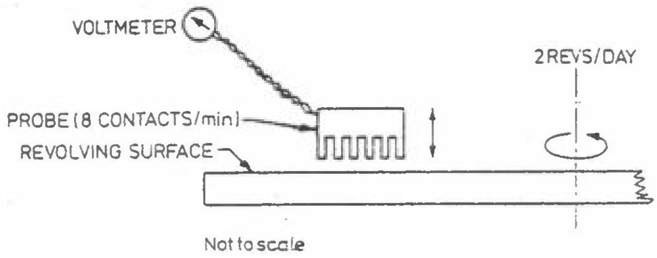

Fig. 2. Schematic diagram of inverted TOW sensor.

The best device to use will depend on the mode of deterioration being studied. As pointed out by Boyd (17), the use of the data may also require other input to describe adequately the responsible ageing environment. For instance, since the freeze-

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POSTBOKS 130, 2001 LILLESTRØM (ELVEGT. 52), NORGE

RAPPORTTYPE Teknisk rapport	RAPPORTNR. TR 17/84	ISBN-8202-7247-544-8	
DATO DESEMBER 1984	ANSV. SIGN. O.F. Skogvold	ANT. SIDER 29	PRIS KR 20,-
TITTEL An electrochemical technique for measurements of time of wetness		PROSJEKTLEDER S.E. Haagenrud	
		NILU PROSJEKT NR. E-8339	
FORFATTER(E) S.E.Haagenrud, J.F. Henriksen, A. Rode og T. Danielsen		TILGJENGELIGHET A	
		OPPDRAGSGIVERS REF.	
OPPDRAGSGIVER (NAVN OG ADRESSE) NILU			
3 STIKKORD (à maks. 20 anslag) Våttidsmålinger	NILU-WETCORR	Cu/Cu sensorer	

REFERAT (maks. 300 anslag, 7 linjer)
En elektrokjemisk teknikk for måling av våttid beskrives. Metoder baseres på måling av strømmen som genereres i elektrolytisk Cu/Cu-celler ved atmosfærisk eksponering. Når denne strømmen overstiger en grenseverdi registreres våttid (TOW). Instrumentet, NILU-WETCORR, som tillater samtidig registrering av 6 sensorer, har helautomatisk datainnsamling og dataanalyse.

TITLE An electrochemical technique for measurements of time of wetness

ABSTRACT (max. 300 characters, 7 lines)
An electrochemical technique for measurement of time of wetness (TOW) is described. The method is based upon measurement of the current generated in electrolytic Cu/Cu celles at the time of atmospheric exposure. When this current exceeds a "wet" threshold value TOW is recorded by the instrument. The instrument which allows for recording of 6 sensors simultaneously is fully automatic with computerized data handling and analyses (NILU-WETCORR).

* Kategorier:	Åpen - kan bestilles fra NILU	A
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