Dataset for the manuscript

**Multisine impedimetric monitoring with an in-depth distribution of relaxation times analysis of WE43 and AZ31 magnesium alloys corrosion**

Maria A. Osipenko(1,2), Jakub Karczewski(1), Marta Prześniak-Welenc(1), Michał Dominów(1), Iryna V. Makarava(1,3,4), Irina Kurilo(2), Dzmitry S. Kharytonau(5,\*), Jacek Ryl(1,\*)

(1) Advanced Materials Center and Institute of Nanotechnology and Materials Engineering, Gdańsk University of Technology, 80-233 Gdańsk, Poland

(2) Department of Physical, Colloid and Analytical Chemistry, Belarusian State Technological University, Minsk 220006, Belarus

(3) School of Engineering Science, Lappeenranta-Lahti University of Technology LUT, Yliopistonkatu 34, FI-53850, Finland

(4) Department of Chemical and Metallurgical Engineering (CMET), School of Chemical Engineering, Aalto University, P.O. Box 12200, FI-00076, Aalto, Finland

(5) Jerzy Haber Institute of Catalysis and Surface Chemistry, Polish Academy of Sciences, Krakow 30-239, Poland

\* corresponding author: [jacek.ryl@pg.edu.pl](mailto:jacek.ryl@pg.edu.pl) (J.R.) [dmitry.kharitonov@ikifp.edu.pl](mailto:dmitry.kharitonov@ikifp.edu.pl) (D.K.)

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Contact Details:

Dzmitry S. Kharytonau

ICSC PAS

ul. Niezapominajek 8

30-239 Krakow, Poland

\*\*\*General Introduction\*\*\*

This dataset contains raw data published in the publication “Multisine impedimetric monitoring with an in-depth distribution of relaxation times analysis of WE43 and AZ31 magnesium alloys corrosion”. Measurement, Volume 222, 2023, 113683, ISSN 0263-2241, https://doi.org/10.1016/j.measurement.2023.113683.

The data was collected at Jerzy Haber Institute of Catalysis and Surface Chemistry, Polish Academy of Sciences (Poland) and Gdansk University of Technology (Poland).

All data are provided under CC0 license.

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\*\*\*Aim of this research\*\*\*

Impedimetric monitoring by Dynamic Electrochemical Impedance Spectroscopy (DEIS) with Dynamic Relaxation Time (DRT) analysis of the kinetics of nonstationary corrosion process of Mg alloys WE43 and AZ31 in Hank’s solution.

\*\*\*Characterization techniques\*\*\*

Dynamic Electrochemical Impedance Spectroscopy (DEIS)

The electrochemical impedance monitoring was performed in a three-electrode setup. A saturated Ag/AgCl electrode was used as the reference electrode, a Pt mesh was used as the counter electrode, and the investigated magnesium alloys were used as the working electrode. The working area was 0.6 cm2. The corrosion cell volume was 45 mL. A heating immersion thermostat (Julabo, Germany) was used to maintain a constant temperature of 37 ◦C. The dynamic electrochemical impedance spectroscopy (DEIS) measurements were carried out in galvanostatic mode at iDC = 0 to imitate open circuit potential (OCP) conditions. The AC perturbation signal was composed of elementary sine waves in the frequency range from 22000 Hz to 7 Hz with 10 points per decade of frequency. The sampling frequency was 128 kHz. Impedance spectra were recorded for 12 h.

Distribution of Relaxation Time (DRT) analysis

Impedance data were analyzed with the DRT method using the DRTTools software [1]. The analysis used a second order regularization derivative with regularization parameter 10-7. In order to cope with the large amount of raw data created by DEIS, an automated algorithmic analysis of the acquired spectra was developed. Preparation and analysis of the data was automated with a Python script using a graphical user interface automation method.

[1] T.H. Wan, M. Saccoccio, C. Chen, F. Ciucci, Influence of the discretization methods on the distribution of relaxation times deconvolution: Implementing radial basis functions with DRTtools, Electrochim. Acta 184 (2015) 483–499, https://doi.org/10.1016/j.electacta.2015.09.097.

Atomic Force Microscopy (AFM)

The surface topography and Volta potential distribution maps were studied with an NTEGRA Aura (NT-MDT) AFM setup in non-contact mode and using HQ:NSC18 probes (MikroMasch) with platinum coating. The Volta potential maps were done using scanning Kelvin probe microscopy (SKPFM) in frequency modulation (FM) mode using an electrically conductive probe with a gold-plated tip.

X-ray photoelectron spectroscopy (XPS)

XPS measurements were performed using an Escalab 250Xi (Thermo Fisher Scientific), with an AlKα X-ray source and spot size 650 μm. The pass energy was 20 eV. The measurements were done under low-energy electron and Ar+ ion bombardment for charge compensation, with a final peak calibration at adventitious carbon C 1 s (284.6 eV). Spectral deconvolution was carried out using the Avantage v5.9921 dedicated software provided by the spectroscope’s manufacturer.

Inductively Coupled Plasma Optical Emission spectroscopy (ICP-OES)

A Perkin Elmer Avio 220 Max ICP-OES was used. A five-point calibration was prepared based on the multi-standard Perkin Elmer – Instrument Calibration Standard 2 with a 100 μg/mL Mg concentration. The specific wavelength for Mg (285.231 nm) was used to determine the magnesium in the samples. The five separate batches were prepared to investigate the magnesium concentration after the 10 min, 1, 3, 6, 12, and 24-hour corrosion processes in HBSS for WE43 and AZ31 samples. One mL of each electrolyte was diluted 10 times into 15-mL centrifuge vials containing 9 mL of 2% HNO3 to obtain an Mg concentration in the measurable, linear range. The total change in the magnesium concentration was determined with respect to the initial concentration of Mg in Hank’s solution.

\*\*\*General description of the data in this data set\*\*\*

Data is divided into 6 folders based on the measurement technique:

AFM – Atomic force microscopy;

DEIS – Dynamic electrochemical impedance spectroscopy;

DRT – Distribution of relaxation time analysis;

ICP-OES – Inductively coupled plasma optical emission spectroscopy;

SEM – Scanning electron microscopy;

XPS – X-ray photoelectron spectroscopy.

\*\*\*Detailed description of the data files\*\*\*

–Folder AFM

This folder contains 2 files, with names:

AZ31 – AFM data for AZ31 Mg alloy;

WE43 – AFM data for WE43 Mg alloy.

Files are presented in format .gwy and can be opened with Gwyddion software.

–Folder DEIS

This folder contains 2 files, with names:

AZ31-zw – DEIS data for AZ31 Mg alloy;

WE43-zw – DEIS data for WE43 Mg alloy.

Files are presented in format .txt and contain all obtained EIS spectra. Each spectrum has two columns: Real (Z’) and Imaginary (Z’’) components of Nyquist plots.

–Folder DRT

The data is divided into two subfolders:

AZ31 – DRT data for AZ31 Mg alloy;

WE43 – DRT data for WE43 Mg alloy.

Folders for each alloy contain 3 subfolders, with names:

Pre-DRT\_Data – contains DEIS spectra selected for analysis;

Post-DRT\_Data – contains results of DRT analysis for each DEIS spectrum from Pre-DRT\_Data folder;

Post-DRT\_Analysis – contains files of extracted impedance parameters from analysis of Post-DRT\_Data.

Files are presented in format .txt.

Each file in folder Pre-DRT\_Data has three columns: frequency (f), Real (Z’), and Imaginary (Z’’) components of Nyquist plots.

Each file in folder Post-DRT\_Data has two columns: Resistance (R) and Tau components of DRT analysis.

Files in folder Post-DRT\_Analysis have two columns: Time (T) and distribution of time constants (tau, files named Constant) or resistance (R, files named Resistance) of processes P1, P2, and P3 obtained from the DRT analysis.

–Folder ICP-OES

This folder contains 1 file ICP-OES, with the results of ICP-OES measurements. File is presented in format .xls and can be opened in Microsoft Excel.

–Folder SEM

This folder contains 2 files, with names:

AZ31 – SEM image for AZ31 Mg alloy;

WE43 – SEM image for WE43 Mg alloy.

Image files are presented in .TIFF format.

–Folder XPS

This folder contains 4 subfolders, with names as follows:

AZ31\_1h – XPS data for AZ31 alloy after 1 h of corrosion in Hank’s solution;

AZ31\_24h – XPS data for AZ31 alloy after 24 h of corrosion in Hank’s solution;

WE43\_1h\_defect – XPS data for WE43 alloy after 1 h of corrosion in Hank’s solution in the defect area;

WE43\_24h – XPS data for WE43 alloy after 24 h of corrosion in Hank’s solution.

Each folder contains survey spectra (name – survey) and files for high-resolution scans of particular elements (name – ElementRegion Scan). Files have .VGD format, which can be opened by XPS proceeding software.