DIAGNOSTICS OF DEGRADATIVE CHANGES IN PARAMAGNETIC ALLOYS WITH THE USE OF LOW FREQUENCY IMPEDANCE SPECTROSCOPY

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Abstract. In the article theoretical bases of the low frequency impedance spectroscopy method and possibilities of its use in the diagnostics of paramagnetic alloys used in aeronautics are explained. The main focus is put on description of interaction of electromagnetic radiation with the material, eddy currents excited in the material (carrier of diagnostic data), modelling of the material and a given diagnostic problem with an equivalent RLC circuit, method of exciting and observation of eddy currents and bases of qualitative and quantitative analysis of the test signal. The knowledge necessary to consciously use eddy currents in NDT and SHM tests directed on the identification of an early phase of material degradation (the phase preceding an open crack) is also of particular importance in the article. The applied measurement instrumentation and sample results of in-house and other research centres’ tests are presented. The in-house tests were performed on objects made of the ASTM 289 class C austenitic steel and ALSi13Mg1CuNi aluminium alloy and on paramagnetic materials used in transport and power industry, whose values of magnetic susceptibility are similar, but their composition, microstructure and other mechanisms of the early phase of fatigue degradation are different. Taking them as an example, the need of taking into consideration the specificity of aeronautical materials and diagnostic problem being solved by the selection of the frequency of electromagnetic radiation, methodology applied in preparation of test methods and diagnostic criteria is highlighted.

Introduction

Safe aircraft operation requires the use of reliable non-destructive methods and monitoring of structural health (NDT & SHM). Modern diagnostics should enable not only to monitor the current structural health but also to detect symptoms of progressing material fatigue in the phase preceding an open crack that enable to prolong the horizon of aircraft safe operation without increasing the frequency of control tests. The above mentioned requirements are not met by typical non-destructive testing methods (VT, UT, ET, MT and RT), whose main task is to detect cracks and big faults/heterogeneity of the structure [1]. The research potential of typical NDT methods is not fully exploited in the scope of detection of the early material fatigue phase, despite the existence of theoretical premises and capabilities of measurement. NDT results (information concerning the current structural health of an object) are rarely used for identification of causes of unusual fatigue changes.
The NDT and SHM are specific in aviation due to complex load spectrum, influence of changing ambient conditions and variety of materials which at the atomic, primitive cell and grain scale differ in: elements, type of chemical bonds and primitive cells. At the micro- and macroscopic scale the differences regard the type and homogeneity of the microstructure. They are reflected by mechanical and physical properties of the material which change under the influence of various mechanisms degradation of the structure [2-7]. For reliable diagnosis of materials necessary to optimize NDT and SHM methods, so that the control activities do not interfere with the main function of the object.

In the article the detection of local anomalies (faults, heterogeneity of the structure, stress concentration areas) in paramagnetic alloys using the low frequency method of impedance spectroscopy is described. The described testing method may be used for diagnosing all electrical conductors, including also ferromagnetic metals.

1. Motivation

For each material there is a close relationship between the chemical composition and microstructure and physical and mechanical properties, e.g. electrical, magnetic, acoustic ones – Figure 1.

Fig. 1. Theoretical premises of the electromagnetic tests in the metal degradation state diagnostics [8]

Quantitative relations depicting the above relationships are subject to change under the influence of [4-7]:

- thermal and mechanical processing,
- material tension,
- phase transitions of the structure,
- operation history,
- ambient conditions (working temperature, temperature of a material during measurements, influence of aggressive atmosphere, erosion, corrosion),
- level of material structure degradation, which is reflected in the following form:
  - increased dislocation density and other microstructure faults and changes in the grain size,
phase changes and occurrence of new structural elements, e.g. carbides – a product of decomposition of a given phase,
- modification of material surface layer properties,
- local plastic deformations at the micro- and macroscopic scale and changes in the structural and magnetic anisotropy,
- occurrence of vacuums, micro- and macrocracks.

During structural degradation of metal the following changes:
- conductivity $\sigma$,
- magnetic permeability $\mu$,
- electrical permittivity $\varepsilon$.

Parameters that may be observed and analysed during electromagnetic tests in radio frequency band from 3 Hz to 3 THz using various test methods and techniques among others, impedance measurements. For metals the observation frequency band may be narrowed to 0 - 100 kHz (ferromagnetic alloys) or 0 - 10 MHz (paramagnetic and ferromagnetic alloys in the state of magnetic saturation) [7, 9].

2. State Observer

The fatigue degradation process of metals is described by a nonlinear simple problem (1) which is a premise for the low frequency electromagnetic testing [7-10] used in the NDT and SHM.

\[
\text{state of the microstructure} \rightarrow \text{electric and magnetic parameters of the material} \quad (1)
\]

In the tests qualitative relations between the impedance $Z_S = Z_S(R_S, L, C, \omega)$ or admittance signal $Y_S = 1/Z_S$ of the measuring probe – state observer element, and frequency response of the following are among others used:
- material impedance, $Z_m = Z_m(\varepsilon, \sigma, \mu, \omega)$ – aim of the tests;
- reference impedance, $Z_0 = Z_0(\omega)$;
- coupling coefficient of the probe with the tested object, $k_m = k_m(\omega, h, PS, M)$, where $h$ is the distance of the probe from the surface of the tested object, PS is a set of parameters describing structural properties of the probe, M is mutual inductance which is described by the following relation:

\[
Z_S = f(Z_m, Z_0, k_m) = f(\underbrace{\omega, h}_{\text{measurement parameters}}, \underbrace{\varepsilon, \sigma, \mu}_{\text{material parameters}}, \underbrace{h/d}, \underbrace{L, R_S, Q, SRF}_{\text{probe parameters}}) \quad (2)
\]

Reliable diagnosis of the material structure condition and its degradation degree requires correct solving of a nonlinear inverse problem [6,7]. For this purpose the following is performed:
1) Measurement of the measuring probe’s impedance response $Z_S(\omega)$ and coupling coefficient $k_m(\omega)$ of the probe with a tested material in selected frequency band $\omega \in (\omega_{min}, \omega_{max})$ using chosen measurement technique [9, 10].
2) Estimation of the frequency response of the material impedance $Z_m(\omega)$.
3) Quantitative and qualitative analysis of the impedance response $Z_m(\omega)$, with the reference impedance response $Z_0(\omega)$. Diagnostic symptoms are identified using the residues method.

\[
\Delta Z(\omega) = \frac{Z_m(\omega) - Z_0(\omega)}{Z_0(\omega)} \quad (3)
\]
Z_0(\omega) most often is the impedance response of the material pattern (initial state of the tested material) or reference material (e.g. dry air), more rarely the structure defect pattern impedance.

4) Analysis of the material nonlinearity degree on the basis of the material impedance distortion response calculated for a constant level of force (voltage or electric current).

\[
THD_\omega = \frac{\sqrt{\sum_{k=2}^{n} |Z(k\cdot\omega)|^2}}{|Z(\omega)|^2}
\]  

(4)

where n - number of the analysed harmonics (most often n = 3 - 6) of the forcing signal frequency

or on the basis of the characteristics of changes in material impedance calculated for a different level of forces

\[
\Delta Z(\omega, \Delta I) = \frac{Z(\omega, I) - Z_0(\omega, I_0)}{Z_0(\omega, I_0)}
\]  

(5)

where I_0 is the reference force level.

5) Diagnostic reasoning - comparison of the material impedance analysis results with the real state of the material structure at the stage of active experiment (laboratory tests and preparation of test methodologies). At the stage of passive experiment (NDT and SHM) the reasoning is performed on the basis of the prepared diagnostic criteria. The test methodology and diagnostic criteria must take account of the influence of boundary conditions (local nonlinearity of the material geometry and changes in shape of a tested material) and material conductivity on penetration of the electromagnetic radiation into the material, real distribution of eddy currents in the material surface layer and dumping of the signal (energy dissipation) in the material.

2.1 Electrical Conductivity of Metal and its Alloys

In general, metals have high electrical conductivity, high thermal conductivity and high density. The electrical properties of a metal represents complex conductivity \(\sigma\) and complex permeability \(\mu\). The electrical properties of metal and dielectric material (nonmetallic parts of structure and some defects e.g. open crack and erosion losses) are also characterised by its complex permittivity \(\varepsilon\). A complex values of \(\varepsilon, \mu, \sigma\) are signalled by:

- the phase shift between electric field \(E\) and current intensity \(J\).
- loss absorption and skin effect

In general, complex conductivity (6) is rank-2 tensors (9 of complex numbers)

\[
\sigma = \sigma_1 + j\sigma_2 = \sigma_{DC} + \omega\varepsilon_2(\omega) + j\omega\varepsilon_1(\omega)
\]  

(6)

The frequency response of conductivity of metals and its alloys is well described by the Drude model (1900) – it is a simplistic model for conduction. The Drude conductivity (7) considers only the conduction electrons in a metal (omits the polarisation of the ion cores, which may occur at the final stage of material degradation) [11].

\[
\sigma = \sigma_1 + j\sigma_2 = \sigma_{DC}/(1 + \omega^2\tau^2) + j\omega\tau\sigma_{DC}/(1 + \omega^2\tau^2)
\]  

(7)

In the 10 MHz frequency band, in which the authors conducted the research, \(\sigma_1 \approx \sigma_{DC}\) and \(\sigma_2 \approx \omega\tau\sigma_{DC}\). The value of the conductivity at the maximum is \(\sigma_1(0) = \sigma_{DC} = \omega_p^2\tau/4\pi\), where \(\omega_p\) and \(\tau\) are respectively the plasma frequency (typically a few THz) and the relaxation time (typically a few hundred femtosecond) in the Drude model of a metal.
2.2 Electrical Model of a Metal

When the electromagnetic radiation wave is many times longer than the size of microstructure elements and plasma frequency \( \omega_p \) in the Drude model, the local and global parameters of a metal may be modelled as a real inductor – Figure 2. The parallel RLC circuit containing 5 elements, in which electric current flows different ways [12]. Besides the branches of inductance \( L \) additionally containing the series resistance \( R_s \) the C capacity branch with the resistance \( R_c \) and resistance branch \( R_p \) occur. The model ensures the correct reflection of the characteristics of impedance \( Z_m \) and admittance \( Y_m \) of a material in the frequency bands used in the low frequency spectroscopy, after adjusting the parameters of the model to the laboratory tests results. The admittance of the equivalent circuit RLC is described by the following equation:

\[
Y_m = Y_R + Y_L + Y_C = \frac{1}{R_p} + \frac{1}{R_s + j\omega L} + \frac{1}{R_c + j\omega C}
\]

(8)

**Fig. 2.** Mapping degradation of the metal in the electrical model (\( t_r \) - the actual service life, \( D \) - the level of structure degradation, \( I_{ec} \) - eddy currents, \( Z_e \) – the electrical impedance, \( Z_m \) – the electromagnetic impedance, \( Z_w \) – the wave impedance) [4, 9]

In low frequency tests it is assumed that all elements of the equivalent circuit may change their values during progressing material degradation. Change in values of at least one of the above mentioned parameters causes a change in impedance and admittance of a material.

\[
Z_m(t) = Z_m(0) + \Delta Z_m(t)
\]

(9)

\[
Y_m(t) = Y_m(0) + \Delta Y_m(t)
\]

(10)

As a result, the following values also change:

- coefficient of the coupling of the probe with a tested material
  \[
  k_m(t) = k_m(0) + \Delta k_m(t)
  \]
  (11)
  by constant distance of the probe from the surface of a tested material \( h \) and unchanging geometry of the probe;

- impedance of the measuring probe
  \[
  Z_z(t) = Z_z(0) + \Delta Z_z(t)
  \]
  (12)
3. Impedance Measurement

For the purpose of assessing the possibility of diagnosing paramagnetic alloys using the low frequency spectroscopy, a series of tests using the RLC IM3532-50 laboratory digital bridge produced by Hioki, handheld UT 612 LCR bridges produced by Uni-T, LDC1000 and LDC1101 evaluation modules produced by Texas Instrument and AD 5933 and AD5934 evaluation module systems produced by Analog Device using PCB surface coils and air coils was performed.

4. Analysis of Measurement Data

For visualisation of measurement results the impedance plane is used - similarly as in the eddy currents NDT. The authors, however, use a reverse arrangement of axes for the purpose of ensuring the proper reflection of the relationship.

\[(R - R_0)/\omega L_0 = f(\omega L/(\omega L_0))\]  

(13)

In the new coordinate system, it is possible to unambiguously assign the value of function to the input value in the frequency band from DC to the resonance frequency of the RLC circuit of the probe and precisely determine frequency responses using multinomial approximation of the measurement data.

For quantitative and qualitative assessment of material degradation changes the relative change of the material impedance \(\Delta Z_m(t)/Z_m(0)\) and its parts \(\Delta R_m(t)/R_m(0)\) and \(\Delta X_m(t)/X_m(0)\) are determined. Changes in parameters of the Drude model \(\Delta \omega_p(t)/\omega_p(0)\) and \(\Delta \tau(t)/\tau(0)\) are also analysed.

The assessment of the parameters of coupling between the coil and a tested material is performed on the basis of reflection coefficient \(\Gamma\) determined on the basis of the equation:

\[\Gamma = \frac{Z_L - Z_0}{Z_L + Z_0}\]  

(14)

in which \(Z_L\) is the impedance of circuit load: coil - tested material, \(Z_0\) is the impedance of reference of dry air or the probe without the influence of a tested material.

5. Results

Some examples of authors test results for paramagnetic alloys aiming at the detection of progressing structure degradation before occurrence of an open crack are presented below. On the basis of measurements and numerical analysis, it was found that using a low frequency impedance spectroscopy and resonance eddy current testing (measurement probe resonance parameters – self resonance frequency, SRF and impedance of parallel resonant circuit, \(R_p\)):

- it is possible to distinguish early stages of material degradation (work hardening, cyclic weakness) – Figure 3;
- it is possible to verify the quality of the microstructure (the quality of material production and supply of spare parts) – Figure 4.

The estimated diagnostic symptoms Par1 and Par2 can be determined on the basis of measurement data from handheld LCR meters (with frequencies of 100 Hz, 120 Hz, 1 kHz, 10 kHz, 100 kHz), and AD5933 and AD5934 impedance converters. Diagnostic symptom of the most sensitive are simultaneous changes in SRF and \(R_p\) – the LCR circuit parameters of the probe determined automatically in low cost LDC1000 and LDC1101 converters.
Fig. 3. Impedance characteristics of probe inductive coupling with austenitic stainless steel before and after the fatigue test ($R_p$ – impedance of parallel LCR circuit, SRF – self resonance frequency of probe coupling with the test material)

Fig. 4. Impedance characteristics of probe inductive coupling with samples of aluminum alloy (samples with different microstructure and the same chemical composition)
Fig. 5. Diagnostic symptoms (estimators Par1 and Par2) in the low frequency band for the data of: a) high cycle fatigue of austenitic steel (data from Fig. 3); b) quality control of the aluminum alloy (data from Fig. 4); dashed lines - the measurement frequency of the UT-612 LCR meters

6. Conclusion

The low frequency impedance spectroscopy provides possibilities to diagnose the degradation degree of the structure of paramagnetic alloys.

Reference