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HR, HU, ID, IL, IN, IR, IS, IT, JO, JP, KE, KG, KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, WS, ZA, ZM, ZW.

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(54) Title: PORTABLE DEVICE FOR MEASURING THE IMPEDANCE SPECTRUM OF STEELS AND METHOD OF MEASUREMENT

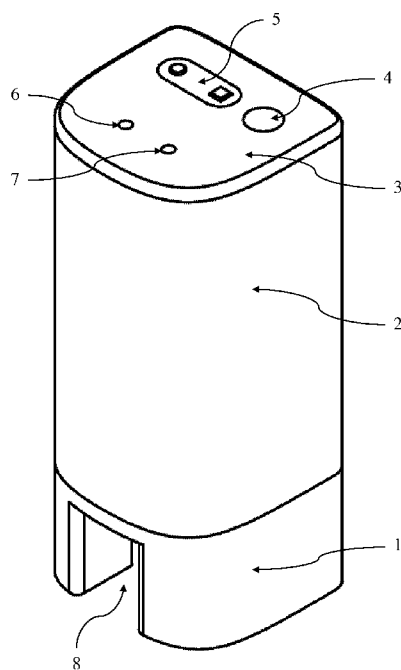


Fig. 1

(57) Abstract: The invention is applied to measure the impedance spectrum for non-destructive diagnosis of steels, in particular heat resistant steels. The invention is intended for in-situ use. The portable device is fixed to the sample to be measured by means of magnets (10, 11) integrated in the magnetic base (1) of the portable device. The magnetic base (1) is adapted to be attached to the surface of the sample to be measured, while exhibiting a cavity (8) for inserting a suitable measuring probe. The inventive method of measurement enables the impedance spectrum to be measured to the depth of the sample to be measured, not just below the surface. The measurement method uses an excitation signal whose amplitude is formed by the superposition of one carrier frequency and at least one harmonic frequency. The impedance is then analysed by parallel synchronous demodulation for each of the frequencies.

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- *in black and white; the international application as filed contained color or greyscale and is available for download from PATENTSCOPE*

Portable device for measuring the impedance spectrum of steels and method of measurement

Field of the invention

The invention relates to the field of non-destructive diagnostics of steels, in particular refractory steels, using impedance spectroscopy.

Background of the invention

The impedance spectrum is the output of an impedance spectroscopy measurement. Impedance spectroscopy is an experimental measurement method that can be used to determine the properties of the system under study, e.g. the structure of the alloy in the surface layer. The measurement method is based on sending a sinusoidal electrical voltage signal through the sample to be measured (e.g. alloy-steel) and then measuring the complex impedance Z as a function of frequency f . The measured results are subsequently evaluated to provide a non-destructive diagnosis of the measured sample.

Diagnostics using impedance spectroscopy is applicable in industry, because the lifetime of components, especially in the energy industry (steam pipelines, etc.) is determined on the basis of a calculation with a significant safety coefficient. With regard to cost optimisation, industrial components can be operated beyond their planned service life if the safety of operation is ensured, e.g. by using non-destructive diagnostic measurements. Currently, the most commonly used methods include total strain measurements on the dies, hardness measurements, microstructure condition assessment by replica sampling, and detection of macro-cracks by magnetic powder method.

The main disadvantages of these established methods include low sensitivity, lack of robustness to adverse operating environments. Furthermore, it is known that replica sampling is a time-consuming method and the microstructure evaluated on the surface often does not correspond to the microstructure immediately below the surface. Modern diagnostic methods include those

based on the principles of micro-indentation, eddy currents and ultrasound. These diagnostic methods are still in the development or pilot deployment phase in the context of the energy industry. The main disadvantages of these methods are their sensitivity only to the state of the microstructure of the surface layer and, in the case of ultrasound, their low sensitivity to changes in the microstructure.

There are inventions US 7 443 177 (B1) and CA 1 235 749 (A), which deal with the use of potential methods in diagnostics, but neither of them addresses the relationship between the electrical impedance spectrum and the state of the microstructure of the material under test.

The object of the invention is to provide a portable device for measuring the impedance spectrum of steels, in particular refractory steels, and a method of measurement which can provide information about the microstructure of the material under test not only in the surface layer but also deeper in the volume of the material within the framework of non-destructive diagnostics.

The subject of the invention

The task is solved by creating a portable device according to the invention described below.

The portable steel impedance spectrum measurement device is made up of a portable device body, which gives the portable device the character of a rigid body, at the same time protects the components of the portable device from damage, and insulates the electrical components of the portable device. Another component of the portable device is a control unit for controlling the operation of the portable device, which is housed within the body. The control unit is a self-contained electronic device that operates according to a software program stored therein and actual user instructions. The portable device further comprises at least one user interface means which is embedded in the body. The user interface means is used to control the portable device by the user and to provide information to the user. Another integral part of the portable device is at least one power supply that distributes electrical power to the electrical components of the

portable device. Last but not least, the portable device includes at least one interconnection cable for connecting the measurement probe.

The subject of the invention consists in that the body of the portable device is provided with a removable magnetic base for fixing the portable device to a sample to be measured. At the same time, a cavity is formed in the magnetic base for connecting a measuring probe. The magnetic base is used to fix the portable device to the sample to be measured so that the relative position between the sample and the electrodes of the measuring probe does not change during the measurement. At the same time, the magnetic base serves as a protective cover for at least part of the measuring probe, which both protects against external influences and protects the user from unwanted contact with the measuring probe. At the same time, it is a very important aspect of the invention that the power supply is a battery. The use of a battery power supply enables the use of the portable device when performing in-situ measurements.

Preferably, the removable magnetic base has two contact surfaces, wherein the first contact surface is adapted to be attached to the body of the portable device, while the second contact surface is adapted to the shape of the sample to be measured. The first contact surface of the magnetic base is for easy attachment and subsequent connection to the body of the portable device. The second contact surface is adapted to the surface of the sample to be measured. The samples to be measured can be pipes with rounded surfaces, steel beams of various shapes, protective casings of silos and containers, etc. The magnetic base and the measuring probe embedded in it must fit properly to the sample to be measured.

It is also advantageous within the invention of the portable device if the control unit includes at least one circuit from the group consisting of an excitation circuit, a circuit for collecting data from an impedance measurement, a circuit for collecting data from a temperature measurement of a sample to be measured, a circuit for managing power supply, a circuit for managing wireless communication with an external device. Integrating these circuits into the control unit, which would otherwise have to be handled separately, positively affects the compactness of the portable device.

It is equally advantageous according to the invention of the portable device that the user interface means is a control button, or a light indicator, or a display, or an audible indicator. The user must be easily but thoroughly informed as to what step of the measurement and what state the portable device is in, at the same time the user must be able to control the operation of the portable device, making audible, light, visual indicators and buttons a most appropriate solution.

Last, but not least, it is advantageous to the invention of the portable device that the interconnecting cable is a six-wire cable. The six-wire cable has the advantage of causing a small parasitic inductive impedance when transmitting electrical signals between the control unit and the measurement probe.

The invention also includes a method of measuring the impedance spectrum of steels, particularly heat resistant steels, for non-destructive diagnosis thereof.

The method of measuring the impedance spectrum of steels, especially heat-resistant steels, consists of a series of consecutive steps:

- a) a measuring probe whose electrodes are in contact with the surface of the sample is applied to the sample to be measured.

The electrodes of the measuring probe are important for ensuring contact with the surface of the sample to be measured and the electrodes of the measuring probe are important for measuring the impedance of the sample to be measured. Each specific implementation of the measuring probe must meet the previous condition.

- b) at least one excitation signal of a sinusoidal waveform is generated in the measuring instrument and introduced via the electrodes of the measuring probe into the sample to be measured.

The sinusoidal waveform of the excitation signal is, in other words, an alternating current waveform. The excitation signal must have known parameters in order to look for correlations between the measured data and the excitation signal parameters. The excitation signal is therefore an alternating electric current with specific parameters.

- c) the measuring instrument records the impedance values of the measured sample for the excitation signal through the measured sample.

Impedance is a physical quantity that, in simplified terms, describes the behaviour of a particular material system when an alternating electric current passes through it. Different material systems have their own behavioural waveforms (impedances) for the same excitation signal.

- d) the measured values of the measured sample are evaluated to determine the properties of the measured sample.

The analysis of the measured impedance values is then used to determine the properties of the measured sample by empirical or analytical methods.

The subject matter of the invention consists in the fact that, in step b), the amplitude of the excitation signal is formed by a superposition of a carrier frequency and at least one harmonic frequency. And further, that in step d) the impedance corresponding to each of the frequencies constituting the excitation signal is evaluated by means of a parallel synchronous demodulation.

Since the excitation signal is composed of several known frequencies simultaneously, it is then possible to recognize in the measured data the individual responses of the material system of the sample to the given frequencies forming a superposition of the amplitude of the excitation signal. For the analysis of the properties of the measured sample, not only the overall picture of the behaviour of the material system to the excitation signal is available, but also a number of other partial pictures. This is advantageous for the reason that the invented method allows the measured sample to be analyzed not only below the surface, but also in depth, since as the depth of penetration of the excitation signal increases, noise increases in the measured data, in which, however, it is possible to trace relevant results for individual harmonic frequencies.

Advantageously, for the invented method, the following relation applies, as the frequency of the excitation signal increases, the measurement is valid for the material system of the measured sample to deeper layers away from the surface.

Practical tests have shown that the invented method works best for steels, especially heat-resistant steels, when the excitation signal has a frequency in the range from 0.1 Hz to 1000 Hz in procedure step b) and when the measuring instrument records impedance values in the range from 0 m Ω to 2 m Ω in procedure step c).

Advantages of the invention include simple attachment and removal from the sample to be measured, the possibility measure with the portable device samples having a rugged surface, the ability of the portable device to allow measurement on a single attachment, and the simple design of the portable device having a low manufacturing cost. A further advantage of the portable device according to the invention is that it allows measurement data to diagnose deeper layers of the sample being measured, not just the subsurface layer. The use of the inventive portable device brings savings from the possibility of safe operation of components in the energy industry even after their planned lifetime.

Brief description of the drawings

The said invention will be explained in more detail in the following drawings, where:

- Figure 1 shows an axonometric top view of the portable device,
- Figure 2 shows an axonometric bottom view of the portable device,
- Fig. 3 shows graphically the waveform of the excitation signal whose amplitude is formed by the superposition of the carrier frequency and of at least one harmonic signal,
- Figure 4 shows a graphical representation of the measured impedance.

Examples of preferred embodiments of the invention

It is understood that the specific embodiments of the invention described and illustrated below are presented for purposes of illustration and not as a limitation of the invention to the examples provided. Those skilled in the art will find or will be able to provide, using routine

experimentation, a greater or lesser number of equivalents to the specific embodiments of the invention described herein.

Figure 1 and Figure 2 show the portable device. The body 2 of the portable device is made of plastic, which is an electrically insulating material, and at the same time the plastic is sufficiently strong and light, and also easy to machine. A removable magnetic base 1 is attached to the body 2 of the portable device from the bottom.

The magnetic base 1 is equipped with two magnets 10 and 11 which do not touch the surface of the sample to be measured. Magnetic base 1 is also made of plastic. The portable device may be equipped with more than one magnetic base 1, the contact surface 9 of which is shaped differently to accommodate samples of differently indented surfaces. The magnetic base 1 has a cavity 8 through which an unshown measuring probe is connected to the portable device. The cavity 8 may accommodate all or part of the measuring probe, the magnetic base 1 forming a protective sheath for the measuring probe or part thereof.

A portable device can be equipped with a variety of measuring probes that vary in design according to the environment in which they are deployed and according to the samples to be measured.

The body 2 of the portable device is provided with a cover 3 on the upper base to protect the internal components of the portable device from the external environment. When folded, the lid 3 and the body 2 form a single body. On the lid 3, which is a removable part of the body of the portable device, there are buttons 5 for manual operation of the portable device, a status indicator 6 and an error indicator 7. In the non-depicted example of the embodiment of the invention, the portable device may be provided with a display, also with touch control capability, or a speaker for reproducing warning and other tones. Further, figure 1 shows a connector 4 for charging the batteries of the portable device.

The control unit consists of a printed circuit board equipped with chips and memories, with integrated circuits in the control unit for wake-up, for collecting data from impedance

measurements, for collecting data from temperature measurements of the measured sample, for power management, for controlling wireless communication with an external device, e.g. with a computer. If not integrated in the control unit, these circuits can be separate electronic components.

The portable device is equipped with batteries for in-situ operation. The batteries can be recharged via the charging connector 4. The control unit operates the batteries.

Figure 3 shows a graphical representation of the excitation signal. The value of the signal current is selected according to the material to be tested and the load of the batteries. The frequency of the excitation signal ranges from 0,1 Hz to 1000 Hz. Figure 3 shows the maximum amplitude and the minimum amplitude, showing the sinusoidal waveform of the signal. The signal shown is not smooth as it is composed of the carrier frequency and of harmonics. The amplitude of the excitation signal is the so-called superposition of these partial frequencies.

When the excitation signal passes through the measured sample, the impedance is measured in the range from 0 m Ω to 2 m Ω . The results of the impedance measurements are sent from the portable device to a computer, where they are immediately stored in a database and subsequently evaluated by analytical and empirical methods. An example of the measured results is shown graphically in Figure 4. The measured results are analysed by synchronous demodulation so that the behaviour of the measured sample can be evaluated separately for each of the excitation frequencies (carrier and at least one harmonic frequency).

Industrial applicability

The portable device and the method of measurement for non-destructive diagnosis of steels according to the invention will find application in the fields of measurement and testing, in particular when performing in-situ diagnostics in the energy industry for structures made of heat-resistant steels.

Overview of relationship tags

- | | |
|----|--|
| 1 | magnetic base |
| 2 | portable device body |
| 3 | lid |
| 4 | charging connector |
| 5 | button for manual control of the portable device |
| 6 | status indicator |
| 7 | error indicator |
| 8 | cavity for measuring probe |
| 9 | contact surface of the magnetic base |
| 10 | magnet |
| 11 | magnet |

PATENT CLAIMS

1. A portable device for measuring the impedance spectrum of steels, comprising a body (2) of the portable device, a control unit for controlling operation of the portable device housed within the body (2), at least one user interface means embedded in the body (2) for and informing the user of the portable device and for the user to operate the portable device, the portable device comprising at least one power supply, and at least one interconnection cable for connecting a measurement probe, **characterized in that** the body (2) is provided with a removable magnetic base (1) for fixing the portable device to the sample to be measured, wherein a cavity (8) is formed in the magnetic base (1) for connecting the measurement probe, and wherein the power supply comprises a battery.
2. The portable device according to claim 1, **characterized in that** the removable magnetic base (1) has two contact surfaces, wherein the first contact surface is adapted to be attached to the body (2) of the portable device, while the second contact surface (9) is adapted to the shape of the sample to be measured.
3. The portable device of claim 1 or 2, **characterized in that** the control unit comprises at least one circuit selected from the group consisting of an excitation circuit, an impedance measurement data acquisition circuit, a temperature measurement data acquisition circuit, a power management circuit, and a wireless communication control circuit for communicating with an external device.
4. The portable device according to any of claims 1 to 3, **characterized in that** the user interface means is a control button (5), or a light indicator (6, 7), or a display, or an audible indicator.
5. A portable device according to any of claims 1 to 4, **characterized in that** interconnecting cable is a six-wire cable.

6. A method of measuring the impedance spectrum of steels, in particular heat-resistant steels, comprising the steps of:
- a) a measuring probe whose electrodes are in contact with the surface of the sample is applied to the sample to be measured,
 - b) at least one excitation signal of a sinusoidal waveform is generated in the measuring instrument and introduced via the electrodes of the measuring probe into the sample to be measured
 - c) the measuring instrument records the impedance values of the measured sample for the excitation signal through the measured sample,
 - d) the measured values of the measurement sample are evaluated to determine the properties of the measurement sample,

characterized in that in step (b) the amplitude of the excitation signal consists of a superposition of the carrier frequency and at least one harmonic frequency, and in step (d) the impedance corresponding to each of the frequencies constituting the excitation signal is evaluated by means of a parallel synchronous demodulation.

7. The method of measurement according to claim 6, **characterized in that** in step b) the relationship applies that the depth of analysis of the properties from the surface layer of the sample to be measured increases with increasing frequency of the excitation signal.
8. The method of measurement according to claim 6 or 7, **characterized in that**, in step (b), the excitation signal has a frequency in the range of 0.1 Hz to 1000 Hz, and wherein, in step (c), the measuring apparatus records impedance values in the range of 0 m Ω to 2 m Ω .

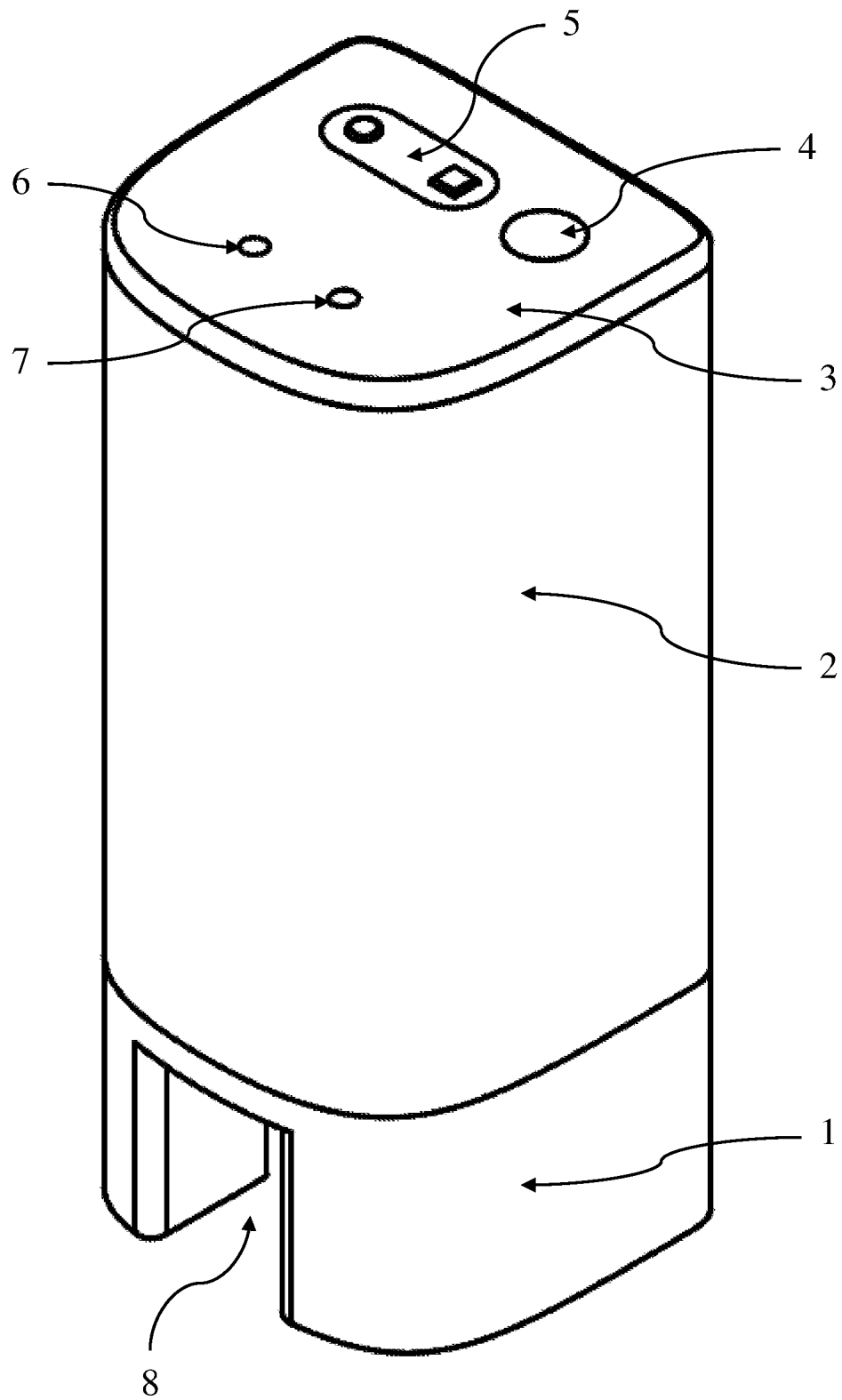


Fig. 1

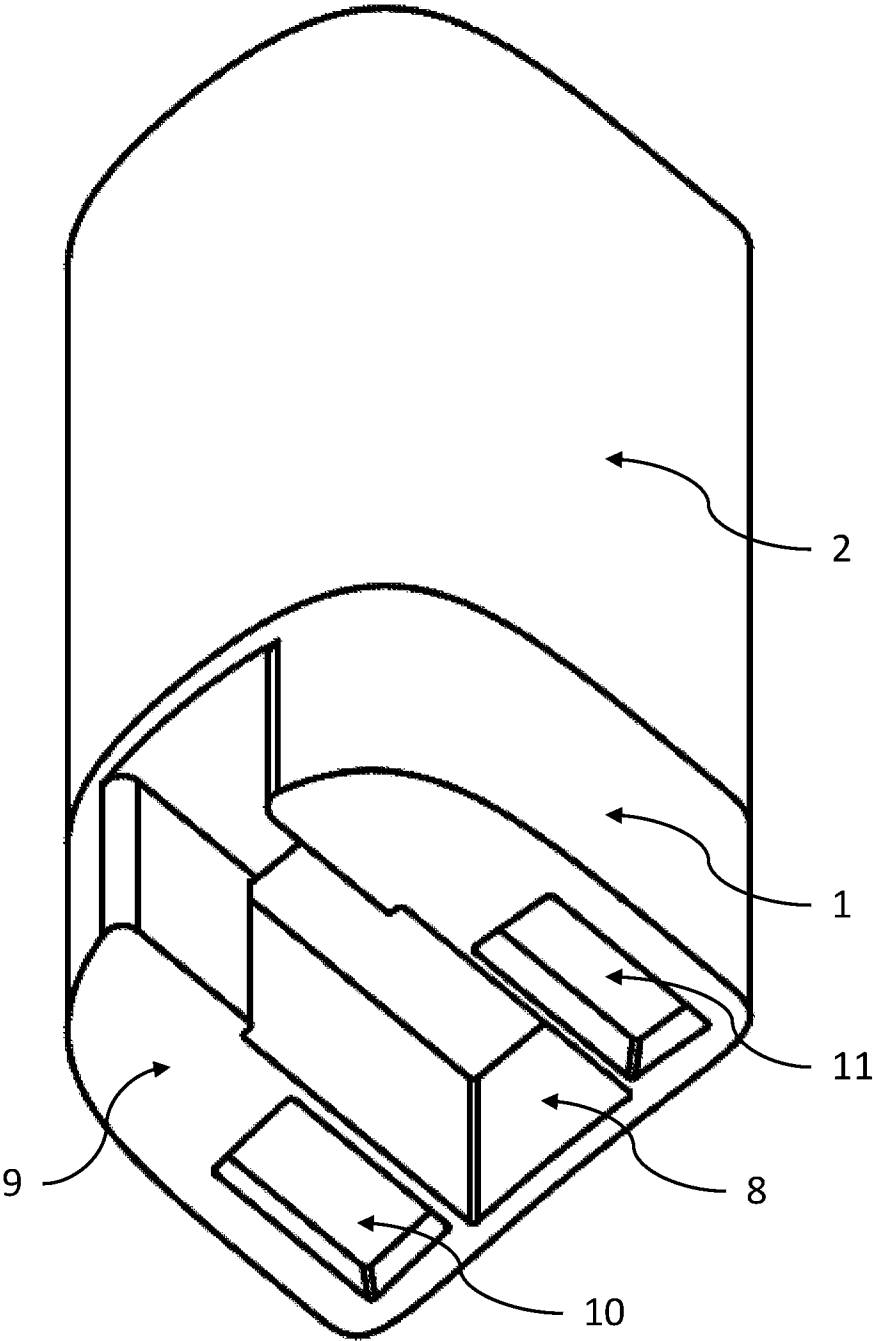


Fig. 2

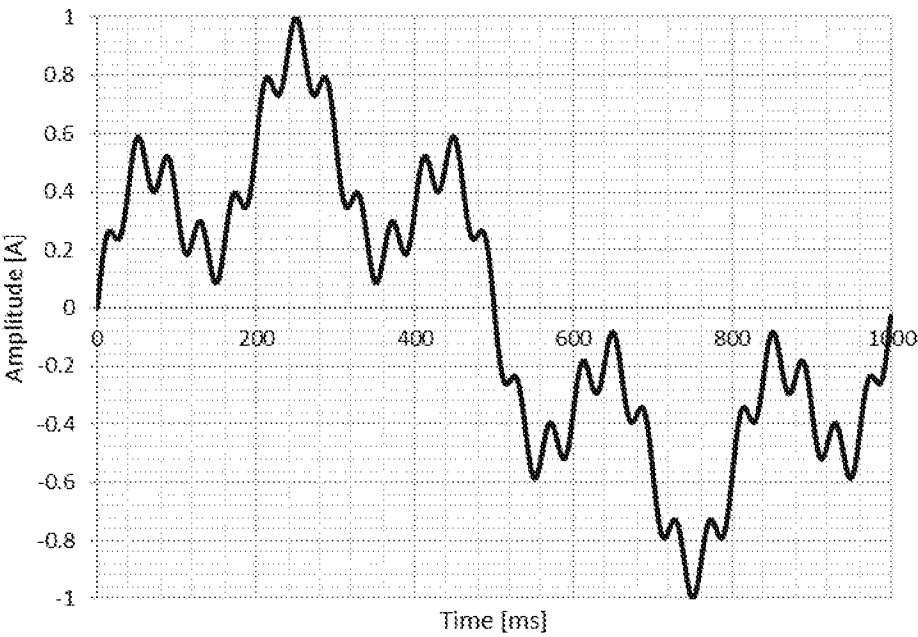


Fig. 3

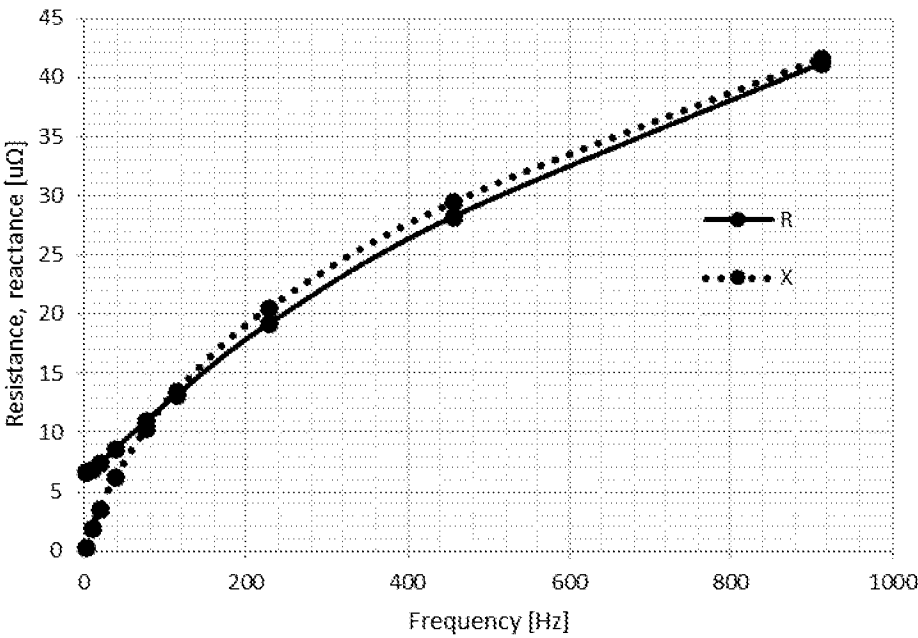


Fig. 4

INTERNATIONAL SEARCH REPORT

International application No
PCT/CZ2021/050106

A. CLASSIFICATION OF SUBJECT MATTER INV. G01N27/02 ADD.		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols) G01N		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EPO-Internal, WPI Data, COMPENDEX, INSPEC		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	TIITTA M ET AL: "Electrical impedance spectroscopy device for measurement of moisture gradients in wood", REVIEW OF SCIENTIFIC INSTRUMENTS, AMERICAN INSTITUTE OF PHYSICS, 2 HUNTINGTON QUADRANGLE, MELVILLE, NY 11747, vol. 73, no. 8, 1 August 2002 (2002-08-01), pages 3093-3100, XP012040172, ISSN: 0034-6748, DOI: 10.1063/1.1485783 paragraph [II.A] figures 1,2,8,9 -----	1-5
Y	WO 2018/093863 A2 (3M INNOVATIVE PROPERTIES CO [US]) 24 May 2018 (2018-05-24) page 7, line 8 - page 8, line 16 figures 1,2 ----- -/-	1-5
☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.		
* Special categories of cited documents : "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family		
Date of the actual completion of the international search 18 May 2022		Date of mailing of the international search report 25/07/2022
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016		Authorized officer Stussi, Elisa

INTERNATIONAL SEARCH REPORT

International application No

PCT/CZ2021/050106

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 7 443 177 B1 (BOWLER NICOLA [US]) 28 October 2008 (2008-10-28) cited in the application column 2, lines 23-25 figures 9,10 column 3, lines 41-48 claim 1 -----	1-5
Y	US 2012/081136 A1 (DAVIS GUY D [US] ET AL) 5 April 2012 (2012-04-05) paragraph [0030] paragraph [0027] figures 3,5 -----	1-5
Y	CN 102 879 028 A (BEIJING INST TECHNOLOGY) 16 January 2013 (2013-01-16) abstract -----	1-5
Y	US 2020/378884 A1 (KHAN ZULFIQAR AHMAD [GB] ET AL) 3 December 2020 (2020-12-03) paragraphs [0020] - [0022] paragraphs [0032], [0033] paragraphs [0065], [0066] claim 16 -----	1-5

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CZ2021/050106

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fees, this Authority did not invite payment of additional fees.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☒ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims;; it is covered by claims Nos.:

1-5

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- ☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- ☐ No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. claims: 1-5

A portable device for measuring the impedance spectrum of steels, comprising a body of the portable device, a control unit for controlling operation of the portable device housed within the body, at least one user interface means embedded in the body for and informing the user of the portable device and for the user to operate the portable device, the portable device comprising at least one power supply, and at least one interconnection cable for connecting a measurement probe, wherein the body is provided with a removable magnetic base for fixing the portable device to the sample to be measured, wherein a cavity is formed in the magnetic base for connecting the measurement probe, and wherein the power supply comprises a battery.

2. claims: 6-8

1.1 A method of measuring the impedance spectrum of steels, in particular heat-resistant steels, comprising the steps of:

- a) a measuring probe whose electrodes are in contact with the surface of the sample is applied to the sample to be measured,
 - b) at least one excitation signal of a sinusoidal waveform is generated in the measuring instrument and introduced via the electrodes of the measuring probe into the sample to be measured
 - c) the measuring instrument records the impedance values of the measured sample for the excitation signal through the measured sample,
 - d) the measured values of the measurement sample are evaluated to determine the properties of the measurement sample, wherein in step (b) the amplitude of the excitation signal consists of a superposition of the carrier frequency and at least one harmonic frequency, and in step (d) the impedance corresponding to each of the frequencies constituting the excitation signal is evaluated by means of a parallel synchronous demodulation.
-

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/CZ2021/050106

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