Research Article

Adam Kondej*, Dominik Kukla, Piotr Wach, and Andrzej Zagórski

Non-destructive method of characterizing nitrided layers in the 42CrMo4 steel using the amplitude-frequency technique of eddy currents

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Abstract: The aim of this work was to investigate the possibility of using the eddy current method, a technique for measuring voltage amplitude and resonant frequency, for non-destructive assessment of the thickness of the near-surface layer of iron nitrides in 42CrMo4 steel after gas nitriding. The scope of the work included preparation of test samples, chemical composition tests, surface roughness measurements, hardness distribution using the Vicker's method and measurements of the thickness of nitrided layer on cross-sections, X-ray phase composition analysis, testing of nitrided layer using the eddy current method, analysis of the correlation of the results of destructive and non-destructive tests. The main research apparatus was the Wirotest M2 with the 25 kHz measuring head. Differences in electromagnetic parameters between the white layer and the rest of the nitrided material, as well as changes in the surface roughness of the layer, are factors influencing the eddy current signal, which allows indirect measurement of its thickness. The analysis of the voltage amplitude is more accurate, than the resonant frequency, in assessing the thickness of nitrides layer. With the increase in thickness of the nitrides layer, the voltage value of the signal of eddy currents increases. The research results also indicate the possibility of using the same measuring head to assess the roughness parameter Ra of the nitrided layer. The Wirotest M2 can be used in quality control of steel parts after nitriding.

Keywords: non-destructive testing, eddy currents, gas nitriding, nitrides zone, thickness measurement, voltage amplitude

1 Introduction

Nitriding is a thermo-chemical treatment of iron alloys, which enhances the mechanical properties of the material (hardness [1,2], resistance against abrasion [3], and material fatigue [4]) and increases its corrosion resistance [5,6]. The process relies on a diffusive saturation of metal surface with nitrogen. Nitriding creates an outer layer with a structure and phase composition that is dependent on temperature, time, and chemical composition of the object and atmosphere [7,8]. It is a process used for treatment of machine parts and tools.

The most common nitriding method used is gas nitriding. Other methods include: plasma nitriding, fluidized bed nitriding, nitriding in powders, and ion nitriding.

Gas nitriding is used on carbon steel, low alloy steel, and alloy steel. Nitriding atmosphere is created from ammonia, a mix of ammonia and dissociated ammonia or a mix of ammonia and nitrogen. This process relies on sustaining the correct thermodynamic activity of the nitriding atmosphere, which is conventionally determined by the value of the nitriding potential.

During the treatment, three basic processes occur: ammonia dissociation, and absorption and diffusion of nitrogen. Nitriding is performed in the temperature range of 420–600°C for several, up to several dozen, hours. The atmosphere parameters are the degree of ammonia dissociation or the nitrogen potential of the nitriding atmosphere. Changes in their values are dictated by the change in composition of inlet atmosphere or the intensity of its flow [9,10].

During the nitriding of ferritic iron or low-carbon steel, there is a possibility of forming of a layer consisting

* **Corresponding author: Adam Kondej,** Łukasiewicz Research Network – Warsaw Institute of Technology (Łukasiewicz – WIT), Warsaw, Poland, e-mail: adam.kondej@wit.lukasiewicz.gov.pl
Dominik Kukla: Łukasiewicz Research Network – Warsaw Institute of Technology (Łukasiewicz – WIT), Warsaw, Poland; Institute of Fundamental Technological Research Polish Academy of Sciences, Warsaw, Poland, e-mail: dominik.kukla@wit.lukasiewicz.gov.pl
Piotr Wach: Łukasiewicz Research Network – Warsaw Institute of Technology (Łukasiewicz – WIT), Warsaw, Poland, e-mail: piotr.wach@wit.lukasiewicz.gov.pl
Andrzej Zagórski: Faculty of Materials Science and Engineering, Warsaw University of Technology, Warsaw, Poland, e-mail: andrzej.zagorski@pw.edu.pl

of three zones: zone α, zones $\gamma' + \alpha$, and zones $\varepsilon + \gamma' + \alpha$. During the nitriding of alloy steel, nitrogen oxides and carbonitrides of alloying elements will be created additionally.

Utility properties, such as resistance to abrasive wear and seizing, corrosion resistance, and fatigue strength are highly dependent on the phase composition. It is presumed that the near-surface zone of iron nitrides $\varepsilon + \gamma'$ increases the resistance to abrasive wear, seizing, and corrosion, while the diffusion zone α increases the fatigue strength [11,12].

Utility properties of the nitrided layer can be enhanced by shaping the proportions of ε and γ' phases in nitrides zone, profiles of nitrogen concentration and hardness in the layer and stress in the layer and core.

The shaping of a phase composition of the nitrides layer is important and requires a precise adjustment of nitrogen supply into steel, and thus the adjustment of kinetics of layer growth.

With the increase in nitriding time in intense nitriding atmosphere, such as ammonia, the thickness of nitrides layer increases, as well as its porosity. Iron nitrides zone is an incremental and partially diffusive layer, and as a result, with the increase in its thickness, the external dimensions of the treated part also increase. This has to be taken into account when dealing with precise parts. The higher porosity of the nitrides layer worsens the hardness and lowers the resistance to corrosion. Thus, it is important to control the thickness of not only the diffusion zone but also the near-surface layer of iron nitrides, which is often omitted during the quality control of finished products [13].

Destructive testing (DT) and non-destructive testing (NDT) are used for examining the created nitrided layers.

The DT consists of metallographic tests on witness samples by using light and electron microscopy [14], hardness measurements on vertical cross-section [15], corrosion tests [16], and X-ray tests [17].

The method of measuring the thickness of the nitrided layer is defined by standards, internal procedures, and customer requirements. The thickness of the nitrides layer is measured directly at the cross section, with a microscope. The thickness of the diffusion zone is indirectly determined by the hardness distribution on the cross section of the sample. Destructive tests are time-consuming and do not allow for the inspection of all parts after the nitriding process.

NDT is a range of research methods and techniques used to determine the condition of the tested material in a non-invasive manner, *i.e.* without disturbing its microstructure or depriving it of its functional properties [18]. The advantage of the NDT is that they can be performed on finished parts. This is especially important when dealing with big, expensive, and important parts, such as moulds and matrices, aviation parts, *etc.* In previous works [19,20], the authors indicate the possibility of using the Barkhausen noise method to assess the thickness of the nitrided layer. Other NDT methods for assessing the thickness of this type of layers are ultrasonic [21,22] and eddy current testing [22]. In the study by Jing *et al.* [23], the acoustic emission and lock-in infrared thermography techniques are used to assess the damage evolution of nitrided steel.

One of the NDT methods, that is already used, to a limited extent, to evaluate the correctness of thermo-chemical treatment implementation is the eddy current testing [22]. This method offers several advantages over others. It is characterized by high sensitivity to various material changes and easy automation of the measurement process. It is fast, gives an immediate response compared to the penetrant method. It does not require a couplant, unlike ultrasonic testing. It allows testing of both ferromagnetic and non-ferromagnetic materials, which is not possible with the magnetic method. Additionally, it is safe for the operator and the environment compared to the radiographic method. The eddy current method allows for testing of conductive materials. If a defect or material change affects the electrical conductivity and/or magnetic permeability of the tested material, it can be detected using eddy currents.

The ε (Fe_3N) and γ' (Fe_4N) phases, forming the nitrides near-surface layer, have different magnetic properties compared to 42CrMo4 steel. ε phase is ferromagnetic with the Curie temperature of 294°C [24]. The room-temperature saturation magnetization of bulk ε is 123 emu/g [24]. γ' compound is also ferromagnetic below 488°C with the saturation magnetization of 186 emu/g at room temperature [24]. The room-temperature saturation magnetization of the 42CrMo4 steel is 191 emu/g [25]. The room-temperature electrical resistivity of nitrides ($\rho \approx$ 140–160 μΩ cm, *e.g.* for Fe_4N ρ = 162 μΩ cm) is greater than that observed in pure iron ($\rho \approx$ 10 μΩ cm) and at least 2–3 times greater than those for high-grade silicon steels ($\rho \approx$ 40–60 μΩ cm) [26]. However, 42CrMo4 steel is characterized by lower resistance at room temperature, which is ρ = 21 μΩ cm [27].

Differences in electromagnetic properties allow us to assume that the thickness of the nitrides layer can be estimated by the eddy current method using the amplitude-frequency technique.

2 The purpose of the work

The purpose of the work is to develop a methodology and implement a control and measurement stand for non-destructive detection of the surface layer of nitrides (white layer) and measurement of its thickness in 42CrMo4 steel after gas nitriding. The implementation will be carried out in Łukasiewicz – WIT in a Centre dealing with thermo-chemical treatment. This implementation will allow for quality control of all parts after the gas nitriding process and for higher quality of provided services.

The scope of the work included the creation of test samples, chemical composition tests, roughness tests, hardness distributions using the Vicker's method, thickness measurements of the nitrided layers on transverse micro-sections, tests of the phase composition of the nitrided layer using the X-ray diffraction method, description and creation of measurement heads, tests of nitrided layers using the eddy current method, analysis of result correlation between DT and NDT.

3 Methodology

The heat-treatable steel samples marked 42CrMo4 (AISI 4140) were the subject of the tests. The samples were cut from a steel rod and had the form of disks with the dimensions Ø38 × 10 mm. The chemical composition of the steel was specified with the use of spark spectrometer GNR S3 MiniLab 300. The average results from three measurements are shown in Table 1. Steel disks were subjected to hardening and tempering.

After heat treatment, the samples were subjected to gas nitriding in the NITREX NX-609 pit furnace. Five processes with varying parameters were performed to obtain samples with different nitrides layer thicknesses, where the diffusion zone thickness exceeds 200 µm. Parameters including temperature, time, and nitriding potential were adjusted (Table 2).

DT was conducted for an unequivocal evaluation of the nitrided layer. From each group, one sample was selected, from which a transverse metallographic sample was made.

Samples were etched using Nital with 2% concentration. Using a light microscope Keyence VHX 5000, a picture of the nitrides layer was recorded with a magnification of 1,000–2,500×, and after that the thickness of the layer was measured in ten places using software. The average values of the thickness of the nitrides layer for individual samples are shown in Table 3.

On a transverse metallographic sample, before etching, the hardness of the core was measured in three places and a hardness distribution was done using the Vicker's method with a load of HV0.5, using an automatic hardness tester Struers DuraScan-70. The thickness of diffusion zone was determined by hardness distribution graph. The value of thickness was defined as the distance from the surface to

Table 1: Chemical composition of 42CrMo4 steel, in weight %

C	Cr	Mn	Si	Cu	Mo	Ni	P	S	W	V	Fe
0.38	0.97	0.78	0.18	0.17	0.16	0.07	0.01	0.01	0.01	0.01	Bal.

Table 2: Technological parameters of the nitriding process

No.	Stage	Parameters of the nitriding process			
		Temperature (°C)	Time (min)	Process atmosphere composition	Nitriding potential*
1	I	520	60	100% NH_3	8.5
	II	520	600	35% NH_3 – 65% NH_{3diss}	0.9
2	I	520	300	100% NH_3	11.0
	II	—	—	—	—
3	I	560	20	100% NH_3	8.5
	II	560	520	30% NH_3 – 70% NH_{3diss}	0.8
4	I	540	120	100% NH_3	17.0
	II	540	1,020	70% NH_3 – 30% NH_{3diss}	1.4
5	I	570	330	100% NH_3	2.5
	II	—	—	—	—

*Nitriding potential N_p – is a parameter that controls and regulates the nitriding atmosphere. N_p determines the quotient of partial pressures NH_3 and H_2 in the atmosphere.

Table 3: List of reference samples

No.	Nitrides zone thickness (μm)	Diffusion zone thickness (μm)	Roughness parameter Ra (μm)
1	7.3	240	0.22
2	10.3	240	0.25
3	14.8	380	0.26
4	20.5	535	0.29
5	25.3	370	0.31

the point where the hardness reaches the core hardness value increased by 50 HV0.5. The values of thickness of diffusion zone for individual reference samples are shown in Table 3.

Measurements of parameter Ra for individual samples were done with portable roughness tester Mitutoyo Surftest SJ-210 in accordance with ISO1997. The length of elementary section lr corresponded with the length of wave λc = 0.8 mm and speed of measurement 0.5 mm/s, and the length of measurement section was 2.5 mm. Ten measurements were done for each sample. The average values of the Ra parameter are shown in Table 3.

The analysis of phase composition of nitrides layer was performed on an X-ray diffractometer Anton Paar XRDynamic 500, equipped with a cobalt lamp (CoKα), in an angle range of 20°–120° and step 0.02° (time per step: 80 s, type of scanning: coupled scan, beam geometry: parallel, detector mode: linear). The phase identification and determining of percentage share of phases were done using SIeve + software suite with PDF-4/Axiom base.

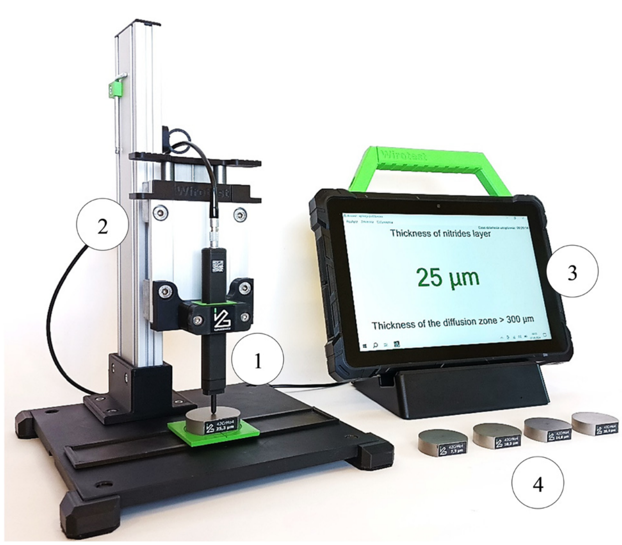

Figure 1: The eddy current measuring system: (1) Wirotest M2 with surface head, (2) holder with guide, (3) tablet with measuring application, and (4) reference samples.

The main method of NDTs of samples after nitriding was the eddy current method. The research equipment consisted of a miniaturized measuring device Wirotest M2, made by Łukasiewicz – WIT, which uses a technique of measuring voltage amplitude and resonance frequency. A holder with guide was designed and made for a repeatable and perpendicular positioning of the head against the testing surface. It provided a continuous pressure of the head. The measuring system is presented in Figure 1. In the preliminary tests, surface heads with the following excitation frequencies were used: 15, 25, 52, 102, 153, 293, 840 kHz, and 1.26 MHz. As a result, the 25 kHz measuring head was selected as the most accurate, *i.e.* for which the highest correlation coefficient of the eddy current signal from the iron nitrides layer thickness was obtained. Then, detailed measurements were carried out for this frequency, the results of which are presented in this study.

4 Results and discussion

In Figure 2, there are microscopic pictures of transverse microsections of five reference samples. The pictures were made with 2,500× magnification and show white layers of different thicknesses (from 7.3 to 25.3 μm). The thickest layer has significant porosity. As the thickness of the iron nitrides layer increases, porosity also increases. For thin layers (<10 μm), it is possible to avoid porosity by applying an appropriate nitriding potential. However, with thicker layers, this becomes challenging, and beyond a certain thickness (>20 μm), it is impossible under industrial conditions. In the case of 42CrMo4 steel, the white layer is homogeneous in structure and thickness, and clearly separated from the diffusion zone. The precipitates visible under the nitrides layer are carbonitrides of alloying elements.

With the increase in nitriding potential, the thickness of white layer increases. With the increase in nitrides layer thickness, the surface roughness parameter Ra also increases (Figure 3).

In Figure 4, the hardness distributions of five samples are shown, based on which the thicknesses of the diffusion zones were determined. No clear correlation was found between the thickness of the near-surface nitrides zone and the diffusion zone. A simultaneous increase in the thickness of the surface nitrides layer and the diffusion zone was observed up to a certain point.

In Figure 5, diffractograms for five reference samples are shown. The presence of two phases: Fe_3N (ε) and Fe_4N (γ') was identified for all layers. The percentage share of individual phases for the given nitrides layer thickness is

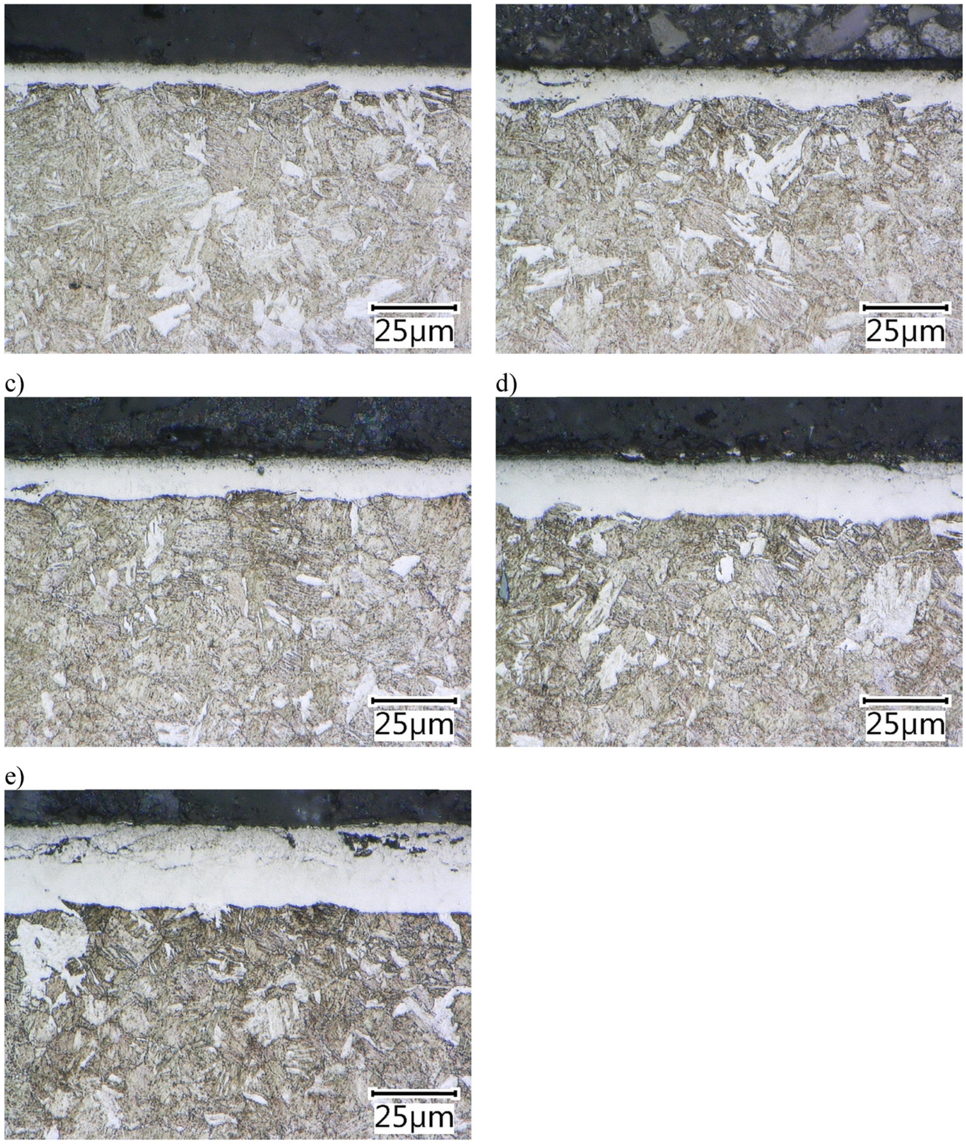

Figure 2: Microscopic images of iron nitrides layers with medium thicknesses: (a) 7.3 µm, (b) 10.3 µm, (c) 14.8 µm, (d) 20.5 µm, and (e) 25.3 µm.

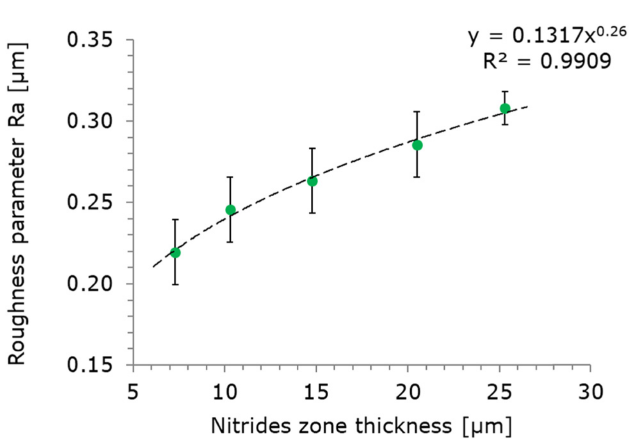

Figure 3: The scatter plot of roughness parameter Ra *vs* nitrides zone thickness.

shown in Table 4. This share does not depend on the thickness of nitrites layer, but on the parameters of gas nitriding process, such as temperature, time and, above all, nitriding potential. In the case of process number 2 (Table 2), due to the high nitriding potential during the process, the content of ε phase was 78% (Table 4). However, in the case of process number 3 (Table 2), which was carried out at a higher temperature but with a low nitriding potential in the second stage, a much lower ε phase content was obtained, the share of which in the near-surface zone was 26%.

In Figures 6–8, there are scatter plots of the dependency of voltage amplitude and resonance frequency on the average thicknesses and roughness parameters Ra of nitrides layers. Plots contain trend lines fitted by using an

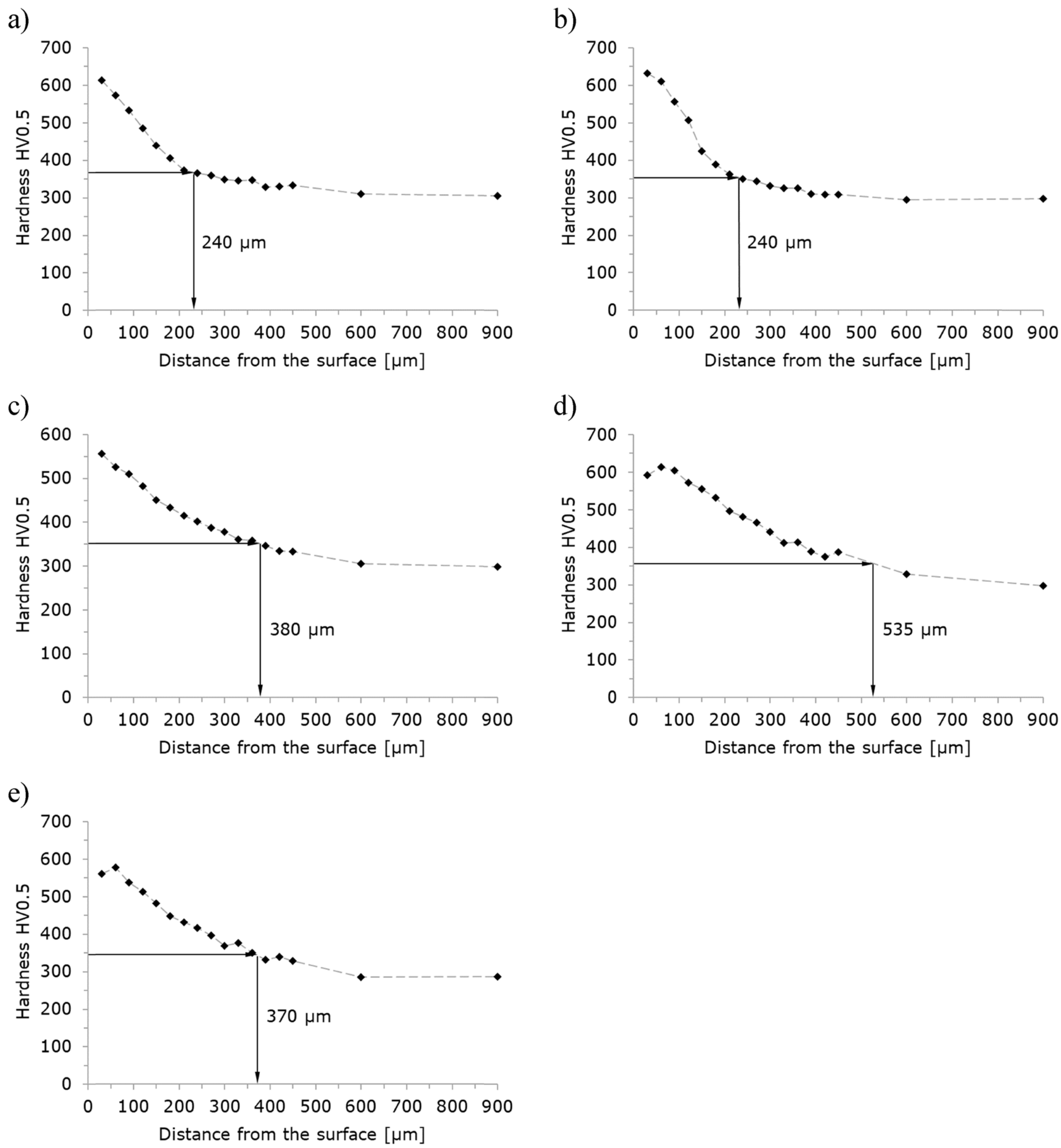

Figure 4: Hardness distributions for samples with a layer of iron nitrides with thicknesses: (a) 7.3 µm, (b) 10.3 µm, (c) 14.8 µm, (d) 20.5 µm, and (e) 25.3 µm.

exponential function, with the equations shown above the dependency curves. Values of the determination coefficient R^2 and the correlation coefficient r are shown in Table 5.

No satisfactory correlation was obtained for the dependence of the eddy current signal on the diffusion zone thickness. To effectively assess the thickness of this zone using the amplitude-frequency technique, it is necessary to choose the optimal operating frequency for the measuring head. Theory suggests that the frequency should be lowered to allow the eddy currents to penetrate deeper and cover an area at a depth greater than the expected thickest diffusion zone. Nonetheless, non-destructive measurement of the diffusion zone thickness poses a challenge. This layer contains a higher concentration of carbonitrides and nitrogen-forming elements, which can affect the eddy current signal. The influence of the steel's chemical composition can significantly impact measurements of the diffusion layer thickness. This issue does not arise when measuring the thickness of the surface layer of iron nitrides in low-alloy steel, which can contain a maximum of two phases: ε and γ'.

The surface roughness of the nitrides layer increases with its thickness. Roughness affects the eddy currents signal, the intensity of which is greatest at the surface. Good correlation was obtained for the dependence of the

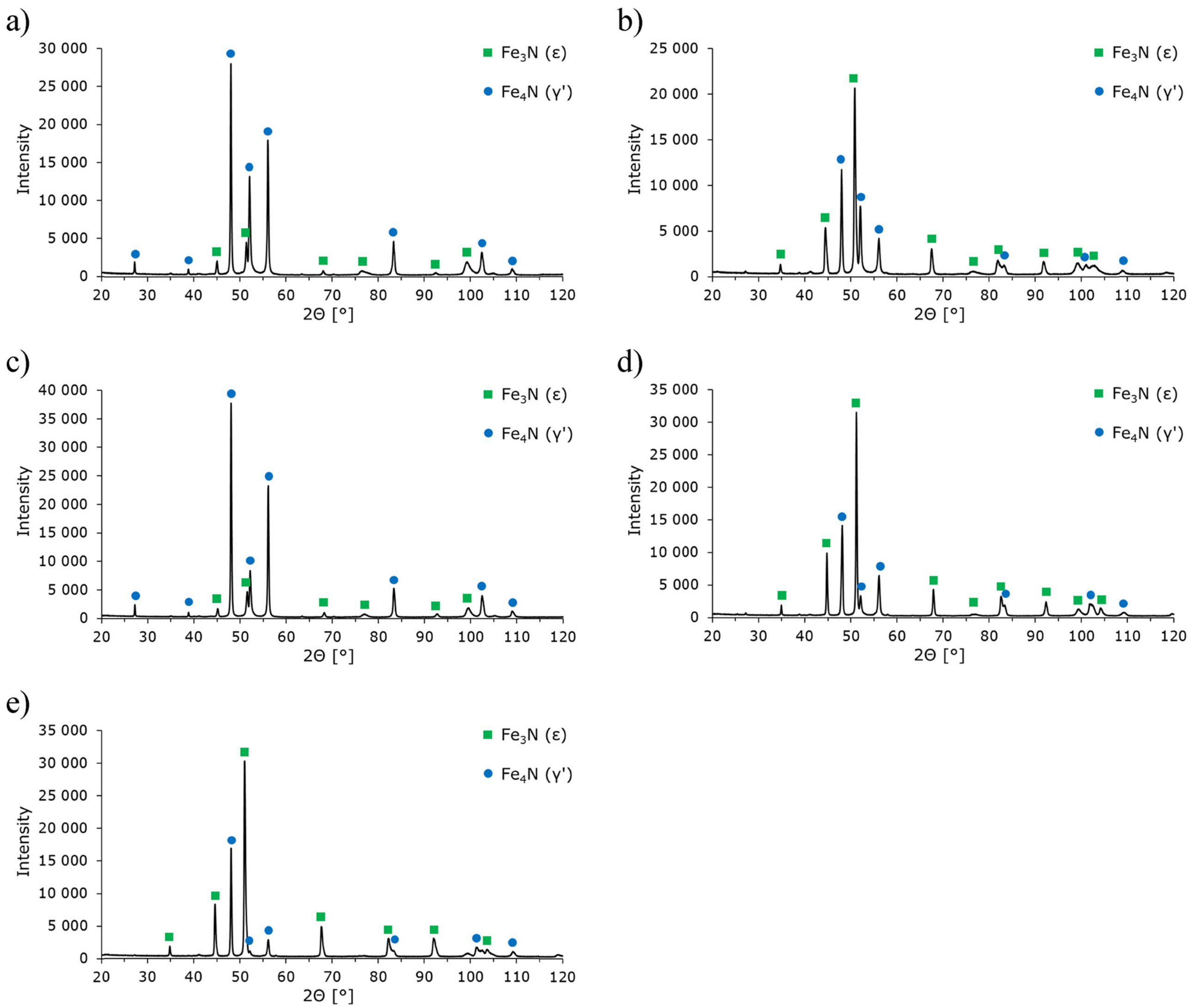

Figure 5: X-ray diffraction patterns for samples with a layer of iron nitrides with thicknesses: (a) 7.3 µm, (b) 10.3 µm, (c) 14.8 µm, (d) 20.5 µm, and (e) 25.3 µm.

Table 4: Percentage of the phases in iron nitrides layers

No.	Thickness of the iron nitrides layer (µm)	The phase percentage (%)	
		Fe_3N (*ε*)	Fe_4N (*γ′*)
1	7.3	29	71
2	10.3	78	22
3	14.8	26	74
4	20.5	73	27
5	25.3	86	14

voltage amplitude on the roughness parameter Ra (Table 5). With the increase in nitrides layer roughness, the voltage amplitude increases (Figure 8a).

Differences in electromagnetic parameters between the white layer and the rest of the nitrided material, as well as changes in the surface roughness of the layer, are factors influencing the eddy current signal, which allows indirect measurement of its thickness. The best correlation was obtained for the dependence of the voltage amplitude on the nitrides layer thickness. For exponential regression, the value of coefficient of determination R^2 was 0.9897. After changing the regression type to linear (Figure 9), the value of coefficient of determination R^2 is also high and equal to 0.9597, which gives the Pearson correlation coefficient r = 0.9796. This indicates a very strong positive correlation between these two parameters. With the increase in thickness of the nitrides layer, the voltage value of the signal of eddy currents increases.

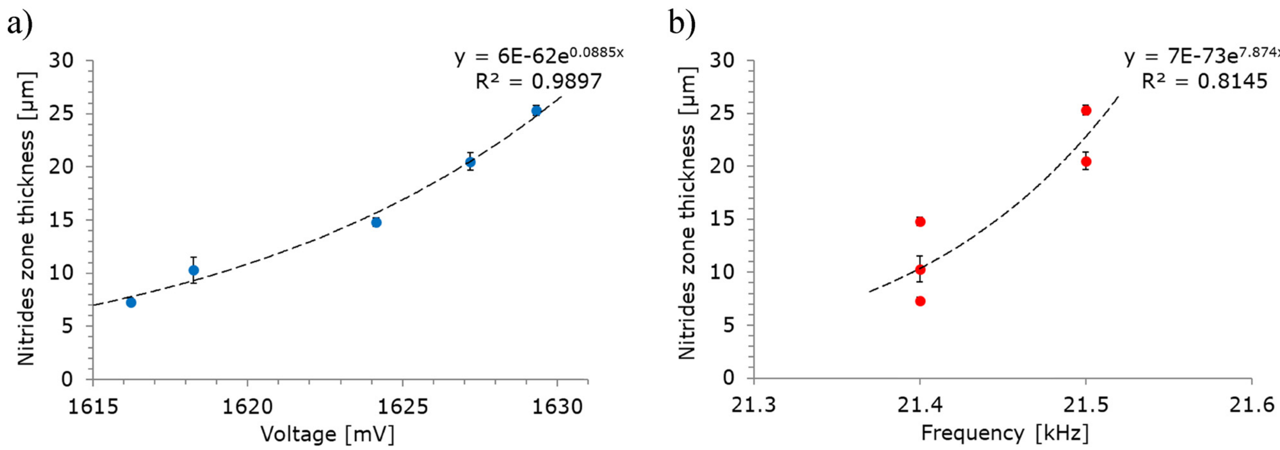

Figure 6: The scatter plot of voltage amplitude (a) and resonance frequency (b) *vs* nitrides zone thickness for the 25 kHz measuring head.

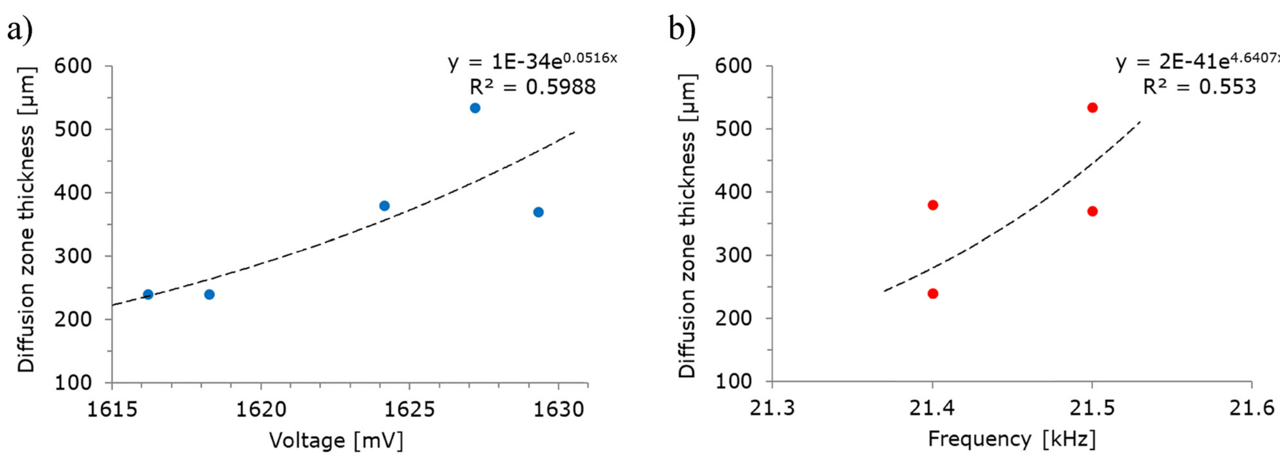

Figure 7: The scatter plot of voltage amplitude (a) and resonance frequency (b) *vs* diffusion zone thickness for the 25 kHz measuring head.

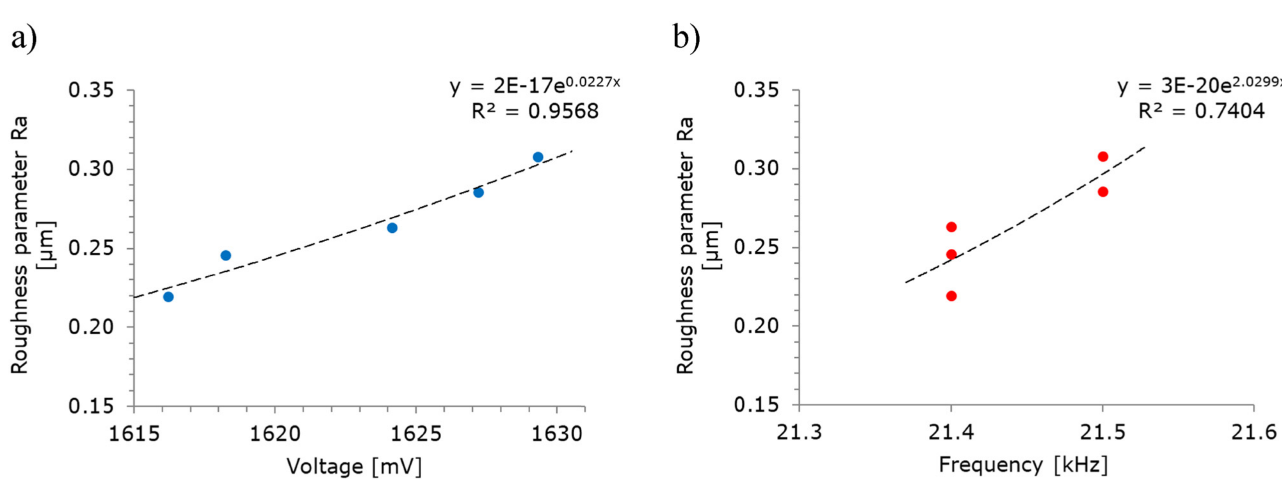

Figure 8: The scatter plot of roughness parameter Ra *vs* voltage amplitude (a) and resonance frequency (b) for the 25 kHz measuring head.

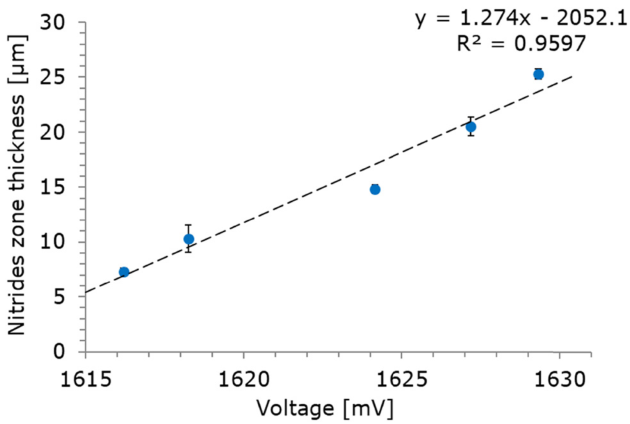

Figure 9: The scatter plot of voltage amplitude *vs* nitrides zone thickness with linear regression.

5 Conclusion

Evaluation of the iron nitrides layer thickness requires selection of the appropriate operating frequency of the measuring head. The depth of eddy current penetration depends on this parameter. As the frequency increases, the depth of eddy current penetration decreases. It is desirable that the induced currents cover the areas being tested in such a way that the indirectly measured quantity has the strongest influence on the eddy current signal.

In the case of eddy current testing of ferromagnets, determining the optimal frequency is difficult because it is necessary to measure the electrical conductivity γ and the relative magnetic permeability μ_r of the tested material. An additional difficulty during testing with the Wirotest M2 device (*i.e.* a resonant system) is that the operating frequency of the head changes as it is brought closer to the tested material. This means that the selection of the optimal frequency is limited to experimental tests. In the future, databases created from these experiments, along with computer simulations, and AI technology may assist in selecting the optimal frequency.

Differences in electromagnetic parameters between the white layer and the rest of the nitrided material are the main factors influencing the eddy current signal. Iron nitrides have lower magnetic saturation and electrical conductivity compared to 42CrMo4 steel. These differences allow for the indirect measurement of the white layer's thickness.

Based on the measurement results, the 25 kHz measuring head was selected as the most accurate for estimating the thickness of the iron nitrides layer in 42CrMo4 steel. The analysis of the voltage amplitude is more accurate, than the resonant frequency, in assessing the thickness of this layer. With the increase in thickness of the nitrides layer, the voltage value of the signal of eddy currents increases.

Roughness affects the eddy currents signal. The surface roughness of the nitrides layer increases with its thickness. The research results indicate the possibility of using the same measuring head to assess the roughness parameter Ra of the nitrided layer. As roughness increases, the amplitude of the eddy current signal also increases. To enhance the correlation coefficient of this relationship, the operating frequency of the measuring head should be increased. This will reduce the depth of penetration of the eddy currents, thereby limiting the influence of the thickness of the nitrides layer and increasing the impact of the roughness parameter on the signal.

Porosity affects the electrical conductivity of the material, which can influence the eddy current signal during the measurement of the iron nitrides layer thickness. This factor was not included in the analysis. Additional studies should be performed to determine the effect of porosity on the eddy current signal.

The produced iron nitrides layers on 42CrMo4 steel had a two-phase structure, regardless of the layer thickness. Depending on the nitriding parameters used, mainly the nitriding potential, different percentage shares of the phases forming the white layer were obtained. This share did not depend on the layer thickness. The results of the conducted studies did not show influence of the proportions of phases ε and γ' on the eddy current signal. Despite these differences, measuring the thickness of the iron nitrides layer is possible using the amplitude technique.

Table 5: Dependency of eddy current parameters on the average thicknesses and roughness parameters of nitrided layers

Dependence	Coefficient of determination R^2
Voltage amplitude – nitrides zone thickness	0.9887
Resonance frequency – nitrides zone thickness	0.8145
Voltage amplitude – diffusion zone thickness	0.5988
Resonance frequency – diffusion zone thickness	0.5530
Voltage amplitude – roughness parameter Ra	0.9568
Resonance frequency – roughness parameter Ra	0.7404

The Wirotest M2 can be used in quality control of steel parts after nitriding to detect the near-surface layer of iron nitrides and to measure its thickness. Its areas of application include tool, automotive, and aviation industries. NDT using the eddy current method allows for the inspection of all manufactured parts and can replace costly and time-consuming DT, which is performed only on selected elements from a given production batch.

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Author contributions: All authors have accepted responsibility for the entire content of this manuscript and consented to its submission to the journal, reviewed all the results, and approved the final version of the manuscript. A.K. designed and manufactured the measurement system, performed DT and NDT on the test samples, and prepared the manuscript with the participation of all co-authors. D.K. designed the methodology for measuring the thickness of the nitrides layer by the eddy current method. P.W. selected the parameters and performed the gas nitriding processes of the test samples. A.Z. designed the methodology for characterizing the reference samples by destructive methods.

Conflict of interest: Authors state no conflict of interest.

Data availability statement: The datasets generated during the current study are available from the corresponding author on reasonable request.

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