NATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY POLITEHNICA OF BUCHAREST
DOCTORAL SCHOOL OF MATERIALS SCIENCE AND ENGINEERING

PhD THESIS SUMMARY

THE USE OF COMPARATIVE METHODS FOR ESTIMATING THE AMOUNT OF RETAINED AUSTENITE IN BEARING STEELS TYPE ASTM A534B29 AND ASTM A485-2

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Table of contents

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ABSTRACT

Bearings used in some application like rail and windmills require keeping the dimensions within strict limits during application. Retained Austenite is a factor influencing dimensional stability of the bearing rings so it should be controlled and maintained within certain limits in bearing steel microstructure. This paper presents some experimental results regarding retained austenite measurement on samples coming from bearing components. Samples made from two steel types were used, both the carburizing steel type ASTM A534B29 and the A485-2 for surface induction hardening, to check the evolution of the retained austenite content measured at different depths. In order to achieve the objective of these works, destructive analyzes were carried out in order to measure the content of retained austenite with the following equipment: optical microscopy, scanning electron microscopy and X-ray diffraction, and on the other hand, the chemical composition was also checked through the optical emission spectrometer but also the hardness profiles on the samples hardened on the surface.

The present paper studies the magneto-inductive method to estimate different retained austenite content in ASTM A534B29 and A485-2 bearing steel while comparing the corresponding measurements by X-ray diffractometry and image analysis by optical microscope. The level of retained austenite content in carburized ASTM A534B29 steel was modified with an additional tempering to yield three samples with distinct levels of retained austenite profiles in the carburized region of the steel. The retained austenite measured at different depths in these samples using the magneto-inductive method had an outcome comparable to other methods. Further discussion based on data suggests that the magneto–induction method yields precise (with an average deviation of 0.5%) results with sufficient sensitivity at different levels (including below 5 vol. %.) of retained austenite. The content of retained austenite in the A485-2 surface hardening bearing steel was modified by only changing the induction hardening temperature, the rest of the process being similar. Due to the content of the significant alloying elements of Chromium and Manganese, paramagnetic carbides were formed which influenced the content of retained austenite evaluated using Feritscope®.Thus, it can be said that this method is feasible for low-alloyed carbon steels, in time what for the alloy steels that form paramagnetic carbides, the method requires improvement.

Keywords: retained austenite; bearing steel; microstructure; non-destructive testing; XRD; magneto–induction method.

CHAPTER 1. INTRODUCTION

Chapter 1, entitled "Introduction" is intended to present the main aspects related to bearing steels, bearing applications, retained austenite in carburized and induction hardened steels and destructive and non-destructive methods used to estimate retained austenite content. Mechanical engineering parts used in demanding applications must be as damage tolerant as possible. Bearings are among the most important steel components in modern machinery, finding their applicability in a wide range of fields such as vehicles, wind turbines and aerospace [1]. The dimensional

stability of bearings became a major issue many decades ago when it was found that bearings containing retained austenite changed dimensionally during storage before being put into service [4]. Measurement of retained austenite content using X-ray diffraction (XRD) is important in quality control and assurance. The role of retained austenite in these microstructures is complex, as it can have both positive and negative effects on the properties and performance of these steels, in terms of component performance and lifetime [24]–[27] . In general, a high content of retained austenite can affect the mechanical properties and in our case the amount of retained austenite can play a significant role in affecting some properties of bearing steels, such as: performance, dimensional stability and longevity of a steel component [28], [29]. The research part consisted of a study on a defined number of carburized and induction-hardened samples, in order to establish a non-destructive control method for estimating the retained austenite (AR) content that would confer the best speed and ease of execution.

CHAPTER 2. THE CURRENT STATE OF RESEARCH IN THE FIELD

Chapter 2, entitled "The current stage of research in the field" is made up of 5 sub-chapters in which the general aspects of bearing steels, the influence of alloying elements on the properties, the retained austenite and its influence on the properties of the bearings are presented, and last but not least are presented the surface improvement treatments applied to the samples that are the subject of this paper.

CHAPTER 3. MATERIALS AND RESEARCH METHODOLOGY

In the experiments in this work, two types of bearing steels were used, for each material type the same analysis methods were used to estimate the amount of retained austenite.

3.1 Bearing steels for carburizing

In order to estimate the content of retained austenite in carburized samples, an ASTM A534B29 steel provided by the Timken company was used. Three cylindrical rollers with a length of 85 mm and respectively a diameter of 48 mm were used for the investigations.

The general flow of the carburizing cycle used for the rolls used as test specimens is presented in Figure 3.2a. All samples of the same dimensions and uniform composition going through the same thermo-kinetics of the carburizing cycle were assumed to have similar RA content (Figure 3.2a). In addition to the carburizing cycle, a few samples were exposed to two hours of secondary tempering at 260 °C and 290 °C to reduce RA content (Figure 3.2b). These tempering temperatures were selected based on studies reporting the effect of tempering temperatures on RA content [65]–[67]. The variation of retained austenite content in heat-treated steels is studied by X-ray diffraction technique, optical microscopy, but also by the non-conventional magneto-inductive method. It is found that the level of retained austenite in the studied steel samples increases with the austenitizing temperature and slowly decreases with the tempering temperature up to 180 °C. Above this temperature RA decreases very rapidly until at

the tempering temperature of 290 °C remains a reduced content of less than 1% (RA). The effects of surface preparation on the results of X-ray diffraction measurements are also demonstrated [68]. Essentially, three samples from distinct rollers were considered for evaluation with specific tempering temperatures as shown in Table 3.1. It should be noted that primary tempering is a final stage of the cementation cycle used for all samples (Figure 3.2a).

Table 3.1 shows the identification of the specimens and the tempering temperatures to which they were exposed.

Tabel 3. 1 Combination of tempering stages exposed to the samples.

Sample #	Tempering Temperatures
1	Primary tempering: 182 °C ± 5 °C
2	Primary tempering: 182 ± 5 °C + Secondary tempering: 260 ± 5 °C
3	Primary tempering: 182 ± 5 °C + Secondary tempering: 290 ± 5 °C

All the samples were evaluated using optical emission spectrometry (OES) to check the composition of the samples as per the standard BS EN ISO 683-17. A Bruker model Q4 TASMAN spectrometer was used to check the chemical composition. In addition, a hardness depth profile was obtained using the Vickers microhardness tester EMCO-TEST at a load of 1 kgf to measure the carburized case depth of the samples.

Tabel 3. 2 Chemical composition (in wt %) of ASTM A534B29 measured by optical emission spectrometrometry

Element	Chemical composition, %wt					
	C	Mn	Si	Cr	Ni	Mo
Measured	0.22 ±0.015	0.69 ± 0.006	0.25 ± 0.010	0.64 ± 0.010	1.61 ± 0.015	0.25 ± 0.012
Standard	0.17–0.23	0.4–0.7	0.15–0.35	0.35–0.65	1.6–2.0	0.2–0.3

The microhardness profile of all samples is presented in Figure 3.3. The effective case depth of the carburized samples is generally measured from the depth immediately below the surface until the Vickers hardness drops to 550 HV. The microhardness profile was performed from the surface to a depth of 4.5 mm. The actual carburized case depth measurements obtained from the hardness profile are shown in Table 3.3. While all samples were subjected to the same carburizing treatment, the effect of the secondary tempering on the actual carburized state of the two samples #2 and #3 is visible. This also indicates the variation of retained austenite as a function of the secondary tempering temperature to which samples #2 and #3 were exposed.

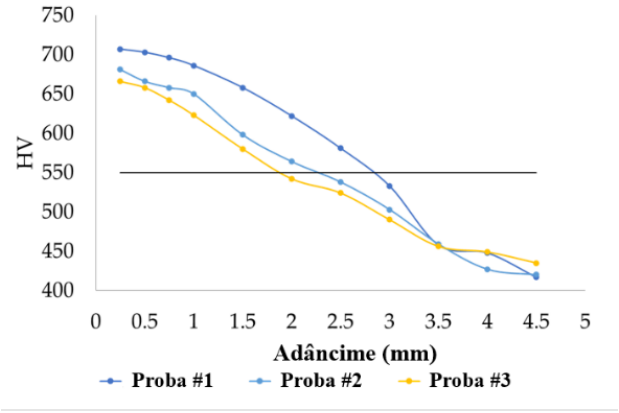


Figure 3. 1 Graphical representation of the e*ffective case carburized depth measurement by microhardness*

The hardness measurements were performed in a straight line perpendicular to the surface of the mounted sample, at regular intervals of 0.25 mm to 1 mm, after which it was continued with a step of 0.5 mm, up to a depth of 4.5 mm depth on each sample, maintaining always a minimum distance of three indentation widths.

Tabel 3. 3 The effective case carburized depth of carburized parts measured by hardness profile

Sample #	Effective case depth [mm]
Sample #1	2.8
Sample #2	2.3
Sample #3	1.9

3.2 Bearing steel for induction hardening

To estimate the amount of retained austenite from induction hardened samples were used bearing steels type A485-2 in the form of linear bars, supplied by the Timken company.

Table 3. 4 Process parameters used for the induction hardening process

Sample #	Temperature Induction Hardening	Temperature Cold treatment	Temperature Tempering
1	810 °C ± 5 °C	-20 °C	182 °C
2	880 °C ± 5 °C	-20 °C	182 °C
3	980 °C ± 5 °C	-20 °C	182 °C

The chemical composition of the bars was verified using optical emission spectrometry as shown in Table 3.6. It was confirmed that the sample was made from A485-2 according to the A485-17 standard [71].

Table 3. 5 Chemical composition (wt%) of A485-2 measured by optical emission spectrometry and according to the A485-17 standard

Element	Chemical composition, %wt					
	C	**Mn**	**Si**	**Cr**	**Ni**	**Mo**
Measured	0.89 ± 0.0.12	1.57 ± 0.021	0.74 ± 0.015	1.58 ± 0.032	0.2 ± 0.015	0.06 ± 0.006
Standard	0.85-1.00	1.40-1.70	0.5-0.8	1.40-1.80	<0.25	<0.10

The microhardness profile of all samples is shown in Figure 3.8. The effective care depth of the induction hardened samples is generally measured starting with the depth immediately below the surface until the hardness drops to an approximate value of 400 HV. The microhardness profile (HV1) was made from 0.5 mm to a depth of 16 mm.

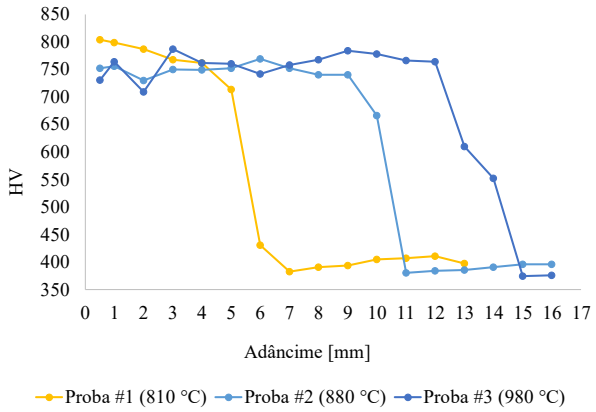


Figure 3. 2 Graphical representation of the microhardness profiles made on each of the 3 samples, to measure the effective induction hardened case depth

Table 3. 6 Effective depth of induction hardened case depth measured using the hardness profile

Sample #	Effective case depth [mm]
Sample #1	5.1
Sample #2	10
Sample #3	12.7

3.3 Estimation of Retained Austenite content in bearing steels

Various methods have been proposed to evaluate retained austenite in steels. There are three main methods for estimating the percentage of retained austenite in steel: optical microscopy, both qualitative and quantitative (OM); X-ray Diffraction (XRD) and Magneto-inductive methods. At the same time, the aim is to estimate a content of retained austenite (RA) of less than 5%, to reduce the uncertainty and the adequacy of the test method according to the practice of the European Union [31], [33]–[35].

The research part consisted of a study on a defined number of carburized and induction hardened samples to establish a method for evaluating retained austenite (RA) by X-ray Diffraction (XRD) that would give the best rapidity and ease of execution. Several sets of samples taken from different steel type with different carbon content were prepared for the study.

The retained austenite evaluated at different depths in these samples using the magneto-inductive method had an equivalent result to other methods described in specialized works. Further discussion based on the data suggests that the magneto-induction method provides accurate results (with an average deviation of 0.5%) with sufficient sensitivity to different levels (including below 5% vol.) of retained austenite.

3.5. The experimental program used in the investigations of this paper

On the two types of material used in the manufacture of bearings (either treated on the surface by carburizing or treated by induction) comparative methods were used: X-ray diffraction, quantitative and qualitative optical metallographic analysis, analysis by magneto-inductive method and analysis by scanning electron microscopy. The experimental program is shown in figure 3.14.

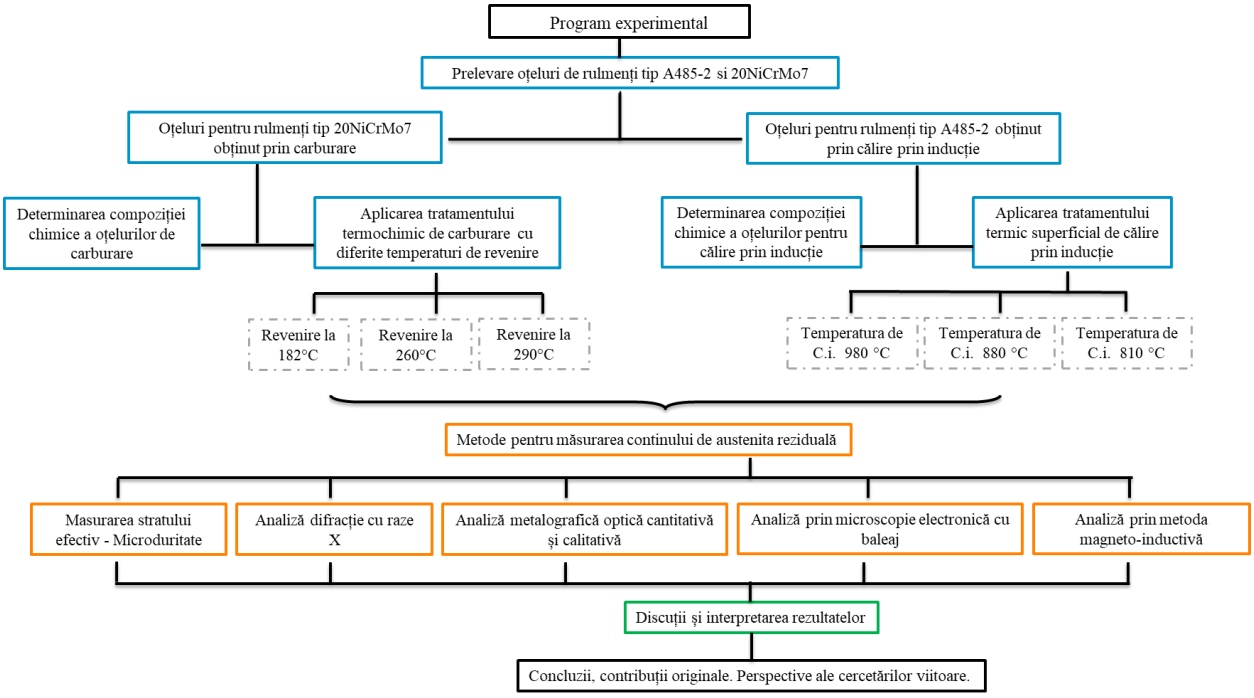


Figure 3. 14 The experimental program carried out within the present work

CHAPTER 4. STUDIES AND EXPERIMENTAL RESEARCH ON THE ESTIMATION OF RETAINED AUSTENITE CONTENT IN BEARING STEELS OBTAINED BY CARBURIZING

4.1 Retained austenite measurement by X-ray diffraction

The results regarding the estimation of retained austenite content by X-ray diffraction are shown in figure 4.1 and table 4.1.

Figure 4 shows representative XRD peaks of martensite and austenite obtained for each sample. It should be noted that the XRD scan was conducted in discrete ranges only to cover the peaks corresponding to martensite and austenite. Under the same operating conditions, the relative change in the peak intensities of martensite and austenite can be noticed among different samples. All four peaks were determined using Cr tube: martensite (211), position 2Θ = 156.3º; martensite (200), position 2Θ = 106º; Austenite (220), position 2Θ = 128º, austenite (200), position 2Θ =79º [86]. Retained austenite (RA) content in vol% was measured based on the relative peak intensities of martensite and austenite at different depths of each sample. These results are presented in Table 4. 1. It should be noted that the uncertainty of the measured retained austenite content is associated with the ASTM E975-13 standard [20].

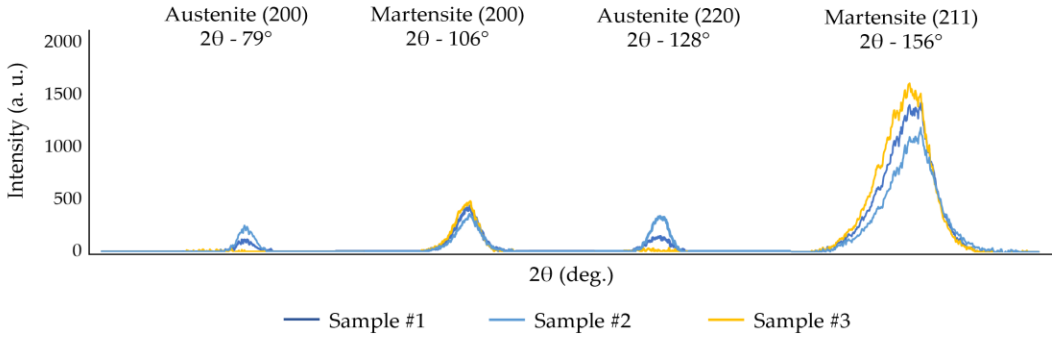


Figura 4. 1 XRD peaks corresponding to austenite and martensite for samples #1, #2, and #3.

Table 4. RA (in vol. %) at different depths measured by XRD.

Depth / Sample #	0 mm	0.2 mm	0.5 mm	1 mm	1.5 mm
Sample #1	25.9 ± 1.9	30.7 ± 2.8	30.9 ± 2.7	21.7 ± 1.6	17.9 ± 1.1
Sample #2	11.4 ± 1.1	10.1 ± 1.0	8.9 ± 0.9	5.1 ± 0.9	3.8 ± 0.3
Sample #3	1.1 ± 0.1	2.1 ± 0.3	2.7 ± 0.4	1.1 ± 0.1	<1.0

Different evolution trends can be observed in the percentage of retained austenite depending on the tempering temperature and the depth of the carburized case (Table 4.1). In sample #1, the average RA content increased from 25.9% to 30.9% up to 0.5 mm and decreased consistently to 17.9% at a depth of 1.5 mm. On the other hand, in the samples tempered at higher temperatures (samples #2 and #3), overall, RA decreased compared to sample #1. For sample #2 (retempered at 260 °C), the average RA content appeared to steadily drop from 11.41% at the surface to 3.8% at 1.5 mm, with a percentage decrease of 66.6%. For sample #3 (retempered at 290 °C), no trend was noted in the RA percentage. The values of RA appeared to fluctuate from the surface to 1.5 mm in a range of 1.08% to 2.7%. However, a slight decrease in average RA values from the depth of 0.5 mm to 1.5 mm can be noticed in sample #3.

4.2 Results regarding the quantitative and qualitative metallographic analysis for estimating the retained austenite content

Locations probed by XRD were parallel investigated using optical microscopy. Figure 4.2 (a), (c) and (e), showing the representative microstructure of all samples, confirmed the presence

of martensite along with austenite and insignificant carbides in each sample. In addition, the representative OM grayscale images of each sample are shown in 4.2 (b), (d) and (f). The appearance of homogeneous martensite (dark) and RA (white) in grayscale allows easier measurement of the individual phase fraction. The effect of varying combinations of primary and secondary tempering treatments in each sample is reflected in grayscale images with varying fractions of RA phase (white). An evaluation made based on the grayscale images to obtain areal % of RA is presented in Table 5. OM-based measurement of retained austenite content at different depths had a similar trend as measured by XRD. However, with this method, it is difficult to accurately quantify a low RA < 5%.

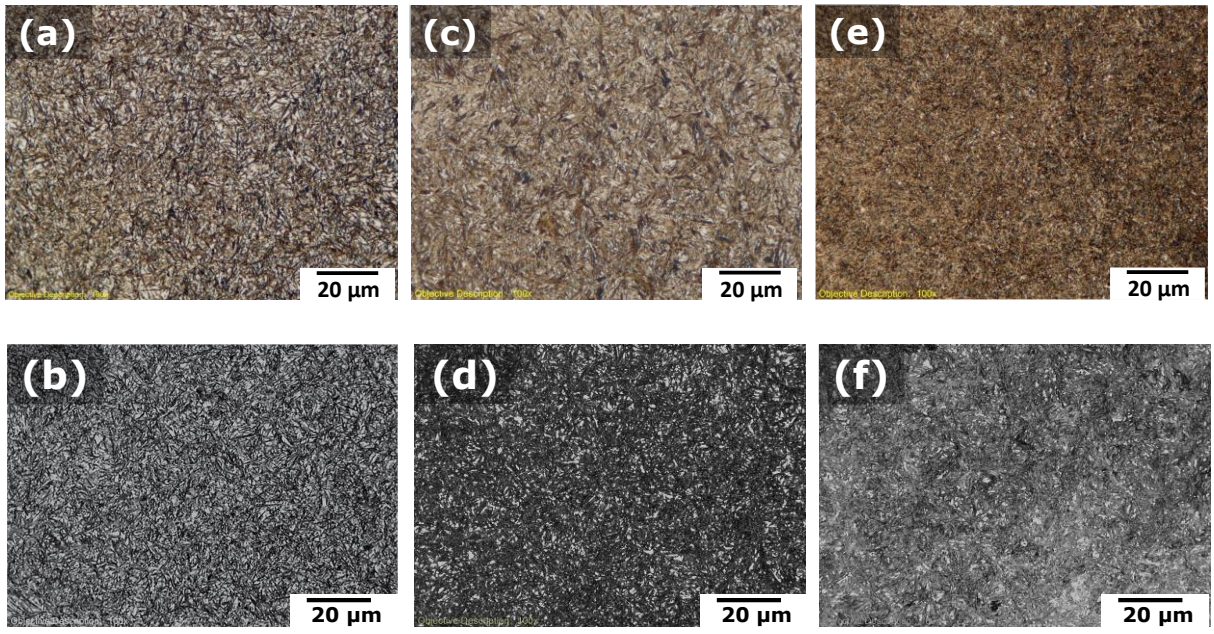


*Figure 4.2. Representative optical micrographs showing presence of martensite and RA at 1000X in sample #1 (**a**), sample #2 (**c**), and sample #3 (**e**); and **(b), (d)** and **(f)** grayscale images of sample #1, #2, and #3, respectively*

Table 4.2. RA (in vol. %) measured at different depths by OM

Depth / Sample #	0 mm	0.2 mm	0.5 mm	1 mm	1.5 mm
Sample #1	27 ± 1.5	28.9 ± 1.1	28.4 ± 1.2	20.8 ± 1.1	12.7 ± 1.5
Sample #2	11.6 ± 0.95	11 ± 1.0	9.4 ± 0.95	<5	<5
Sample #3	<5	<5	<5	<5	<5

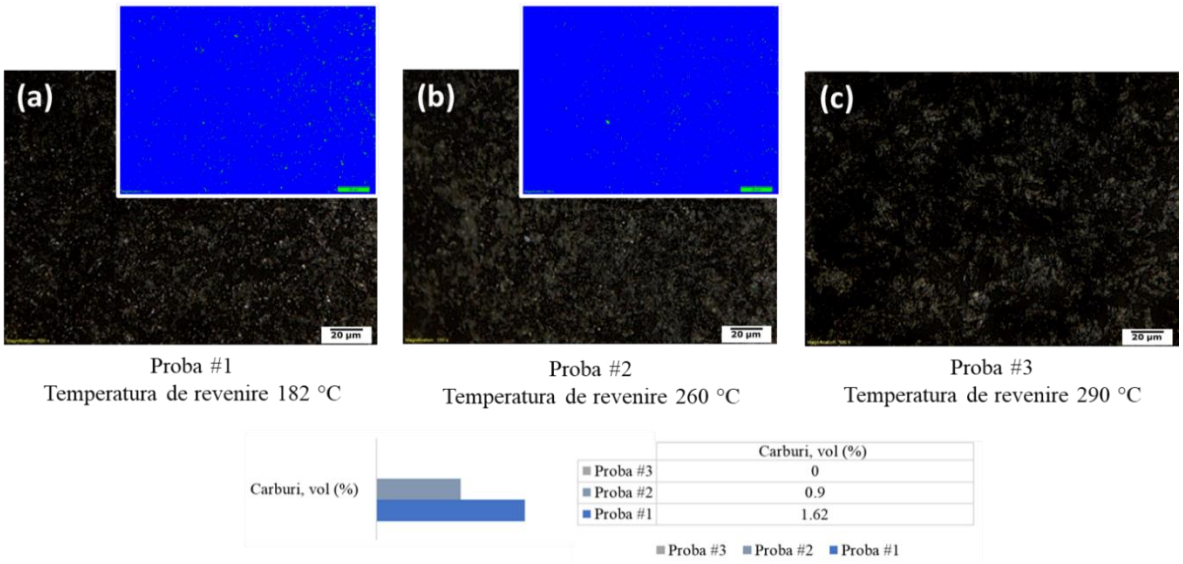


Figure 4. 5. Representative microstructures for carburized ASTM A534B29 steel showing the presence of an insignificant amount of carbides in sample #1 (a), sample #2 (b), and sample #3 (c) at 1000X

Although the optical micrographs did not reveal the presence of a considerable fraction of carbides that would affect the measurement of the retained austenite fraction, the samples were etched to obtain a sufficiently good contrast favoring the highlighting of the carbides. In order to be able to estimate the volume fraction of carbides, the images were processed with ImageJ software. The results presented on the graph in figure 4.5 confirm that the percentage of carbides present in the microstructures is insignificant, which made it favorable to estimate the content of retained austenite using the magneto-inductive method.

However, the low content of Cr (0.64 %) and Mo (0.25 %) in the ASTM A534B29 steel grade cannot form a significant amount of carbides to affect the result of the magneto-inductive method estimates. Thus, the iron carbide fraction is expected to be higher than the rest of the carbides. Within the limits of the visual sensitivity of optical microscopy, no carbide fraction was detected at a given depth. However, there is the possibility of a minimal volume fraction (below 2%) of paramagnetic carbides.

4.3 Results regarding the estimation of retained austenite content by magneto-inductive method

Figure 4.6 shows the comparison of the measured (RA_m) and actual/true (RA_T) RA (in vol. %) at the surface of the samples considered in the present work. As mentioned in Section 3.3, the difference between RA_m and RA_T arises due to the convex curvature of the roller, which leads to non-uniform spacing between the probe and the surface of the sample. The average actual/true RA value appeared to be less than the measured RA value within the sensitivity limits of Feritscope®.

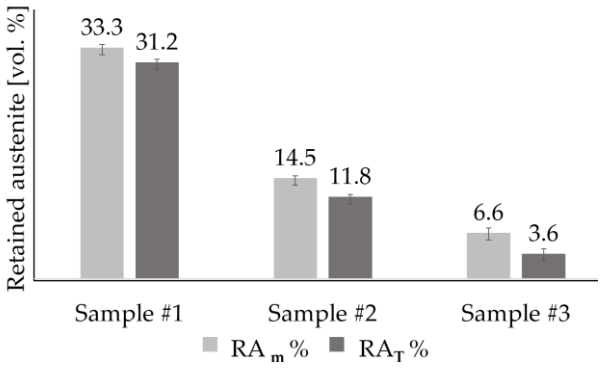


Figura 4. 2 Measured and actual content of RA (in vol. %) by magneto-induction method

The real retained austenite (RA_T) values measured by the magneto–induction method at the surface is close to the values obtained by X-ray diffraction and image analysis (Figure 4.2). As mentioned earlier, the correction factor was not considered at a depth other than the surface as the curvature effect is insignificant after electropolishing. Thus, instrumentally measured values of RA at different depths for all samples are presented in Table 6. It can be noticed that the values of RA measured at different depths are comparable to those obtained by XRD and optical microscopy. In addition, the insignificant deviation of the RA values measured by Feritscope® indicates a precise measurement of RA compared to XRD. It appears that Feritscope® is adequately sensitive to detect the RA below 5% with values compared to those obtained by XRD.

Table 4.3 RA (in vol. %) measured at different depths by magneto-induction method

Depth / Sample #	0 mm	0.2 mm	0.5 mm	1 mm	1.5 mm
Sample #1	31.2 ± 0.4	29.8 ± 0.5	29.1 ± 0.7	23.9 ± 0.4	21.5 ± 0.4
Sample #2	11.8 ± 0.4	11.5 ± 0.5	10.1 ± 0.5	9.7 ± 0.5	6.2 ± 0.5
Sample #3	3.6 ± 0.8	3.4 ± 0.6	4.4 ± 0.7	2.9 ± 0.5	1.5 ± 0.4

Comparative analysis of the results

In order to compare the results provided by each method, the measured RA values are summarized for easier visual comparison in Figure 4.7. It should be noted that for samples #2 and #3, the immeasurable low RA values measured by optical microscope and XRD have not been included in the plot. This suggests the inadequate sensitivity of these methods for low RA content. On the other hand, measurement via the magneto–induction method is possible at different levels of RA. In addition, Feritscope® is an easy and quick tool to get consistent results that are in good agreement with those obtained by XRD and optical image analysis. The following discussion focuses on the comparison of each technique with other techniques in detail.

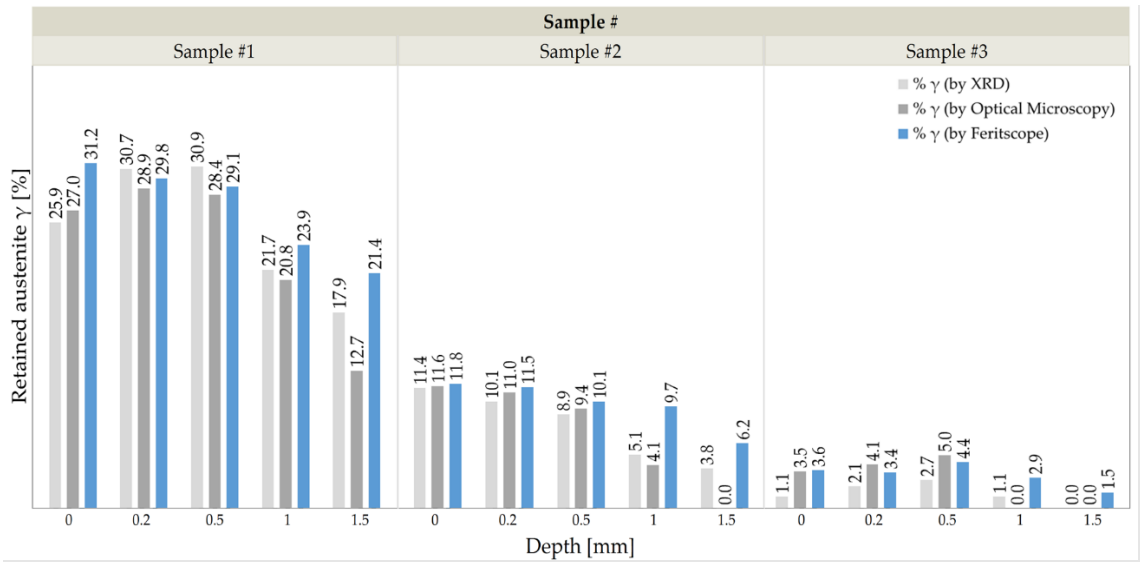


Figura 4.7 Comparison of RA values measured by all three methods: Feritscope®, XRD, and quantitative image analysis using optical micrographs.

Tabel 4. 4 Results of retained austenite content estimated by methods: X-ray diffraction (XRD), quantitative image analysis using optical micrographs and Feritscope®

Method	Depth / Sample #	0 mm	0.2 mm	0.5 mm	1 mm	1.5 mm
XRD	Sample #1	25.9 ± 1.9	30.7 ± 2.8	30.9 ± 2.7	21.7 ± 1.6	17.9 ± 1.1
	Sample #2	11.4 ± 1.1	10.1 ± 1.0	8.9 ± 0.9	5.1 ± 0.9	3.8 ± 0.3
	Sample #3	1.1 ± 0.1	2.1 ± 0.3	2.7 ± 0.4	1.1 ± 0.1	<1.0
OM	Sample #1	27 ± 1.5	28.9 ± 1.1	28.4 ± 1.2	20.8 ± 1.1	12.7 ± 1.7
	Sample #2	11.6 ± 0.9	11 ± 1.0	9.4 ± 1.5	<5	<5
	Sample #3	<5	<5	<5	<5	<5
Feritscope®	Sample #1	31.2 ± 0.4	29.8 ± 0.5	29.1 ± 0.7	23.9 ± 0.4	21.5 ± 0.4
	Sample #2	11.8 ± 0.4	11.5 ± 0.5	10.1 ± 0.5	9.7 ± 0.5	6.2 ± 0.5
	Sample #3	3.6 ± 0.8	3.4 ± 0.6	4.4 ± 0.7	2.9 ± 0.5	1.5 ± 0.4

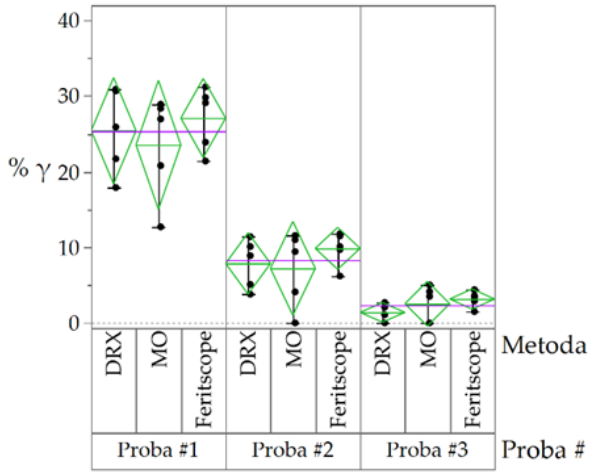


Figure 4. 9 Statistical analysis of retained austenite content estimation using destructive methods (XRD and OM) and magneto-inductive method (Feritscope®)

Table 4. 5 Average values and standard deviations calculated to facilitate comparison of retained austenite results estimated by XRD, OM and Feritscope® methods

Sample#	Method	Nr.	Mean	Standard Deviation
#1	Feritscope®	5	27.08	4.21
	OM	5	23.54	6.87
	XRD	5	25.41	5.66
#2	Feritscope®	5	9.86	2.23
	OM	5	7.22	5
	XRD	5	7.86	3.27
#3	Feritscope®	5	3.16	1.07
	OM	5	2.54	2.38
	XRD	5	1.39	1.04

As we can see, the technique gives very promising results to estimate the content of retained austenite (RA%) in the carburized samples produced from this steel type. Analysis of Variance (ANOVA), is a statistical method that was conducted to compare the means between the results provided by Feritscope® compared to XRD and OM. We can conclude that each equipment estimated similar values of retained austenite content. The final average of the values is represented by the average of the 5 evaluations obtained at different depths at which the determinations were made: on the surface, 0.2 mm, 0.5 mm, 1 mm and 1.5 mm. The highest value of the standard deviation was found to be given by the results estimated by the metallographic analysis.

In addition, the deviation of the retained austenite content (RA) values estimated by Feritscope® indicates the precise measurement of the RA which is compared with the results obtained by XRD. Feritscope® appears to be sensitive to estimating RA content below 5%.

CHAPTER 5. STUDIES AND EXPERIMENTAL RESEARCH ON THE ESTIMATION OF RETAINED AUSTENITE CONTENT IN BEARING STEELS OBTAINED BY INDUCTION HARDENING

5.1 Retained austenite measurement by X-ray diffraction

The results regarding the estimation of retained austenite content of bearing steels heat-treated by induction hardening by X-ray diffraction are shown in figure 5.1 and table 5.1.

Figure 5.1 represents the representative X-ray diffraction (XRD) peaks of martensite and austenite obtained for each sample. It should be noted that the X-ray diffractometer (XRD) scan was performed at discrete intervals only to cover the peaks corresponding to martensite and austenite. Under the same operating conditions, the relative change in peak intensities of martensite and austenite can be observed between different samples.

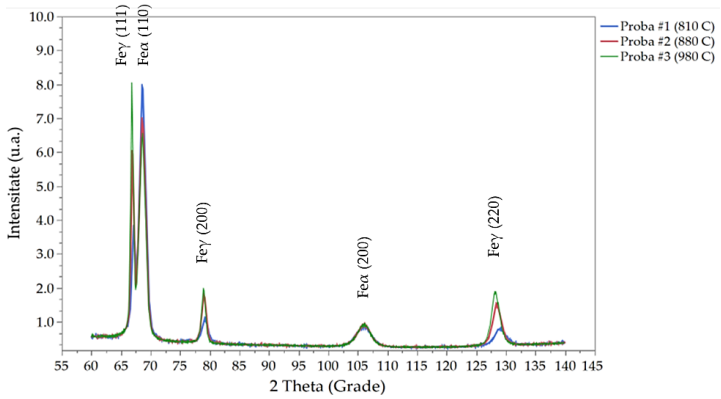


Figure 5. 1 XRD diffractogram corresponding to retained austenite and martensite for the experimental samples induction hardened at 810 ℃, 880 ℃ și 980 ℃

Table 5. 1 The amount of retained austenite (in vol. %) measured by X-ray diffraction analysis, at different depths in the induction hardened samples

Sample # / Depth [mm]	Sample #1 (I.H. at 810°C)	Sample #2 (I.H. at 880°C)	Sample #3 (I.H. at 980°C)
0.05	23.3 ± 1.4	33.1 ± 1.4	34.4 ± 3.1
0.2	21.7 ± 1.5	33.9 ± 1.2	39.4 ± 1.2
0.5	20.0 ± 0.8	35.6 ± 1.1	35.2 ± 2.5
1	17.9 ± 0.9	33.4 ± 1.9	31.4 ± 1.7
1.5	16.6 ± 1.6	32.1 ± 1.2	33.0 ± 2.0
2	16.5 ± 0.8	30.2 ± 1.5	33.2 ± 0.5

From the analysis of the values presented in table 5.1, different trends in the evolution of the percentage of retained austenite can be observed depending on the temperature at which the experimental samples were induction hardened. In sample #1, the average RA content decreased from 23.3% to 20.0% up to 0.5 mm, with a constant decreasing trend to 16.5% at a depth of 2 mm.

On the other hand, in the samples that were heat-treated at a higher temperature (samples #2 and #3), the proportion of general RA decreased in a smaller percentage compared to sample #1.

5.2 Quantitative and qualitative metallographic analysis results for estimating retained austenite content

The results regarding the estimation of the retained austenite in the bearing steels heat-treated by induction are shown in figures 5.3-5.20 where we can observe the characteristic microstructures of the sample but also the images processed in grayscale to be able to quantify the retained austenite content and the percentages obtained with using the software are shown detailed in table 5.2.

The locations evaluated by X-ray diffraction (XRD) were investigated in parallel using optical, quantitative and qualitative metallography analysis. Figure 5.2 (a–c) shows the visual appearance of representative microstructures of the samples at 200x magnification, which confirmed the presence of an inhomogeneous martensitic microstructure together with retained austenite and carbides. At higher magnification, depending on the temperatures at which they were subjected to the process, either segregations of carbides in the case of the sample hardened superficially at 810 °C, or segregations of retained austenite in the case of the other two samples #2 and #3 could beobserved. The microstructures were made at a magnification of 200x and 1000x, respectively.

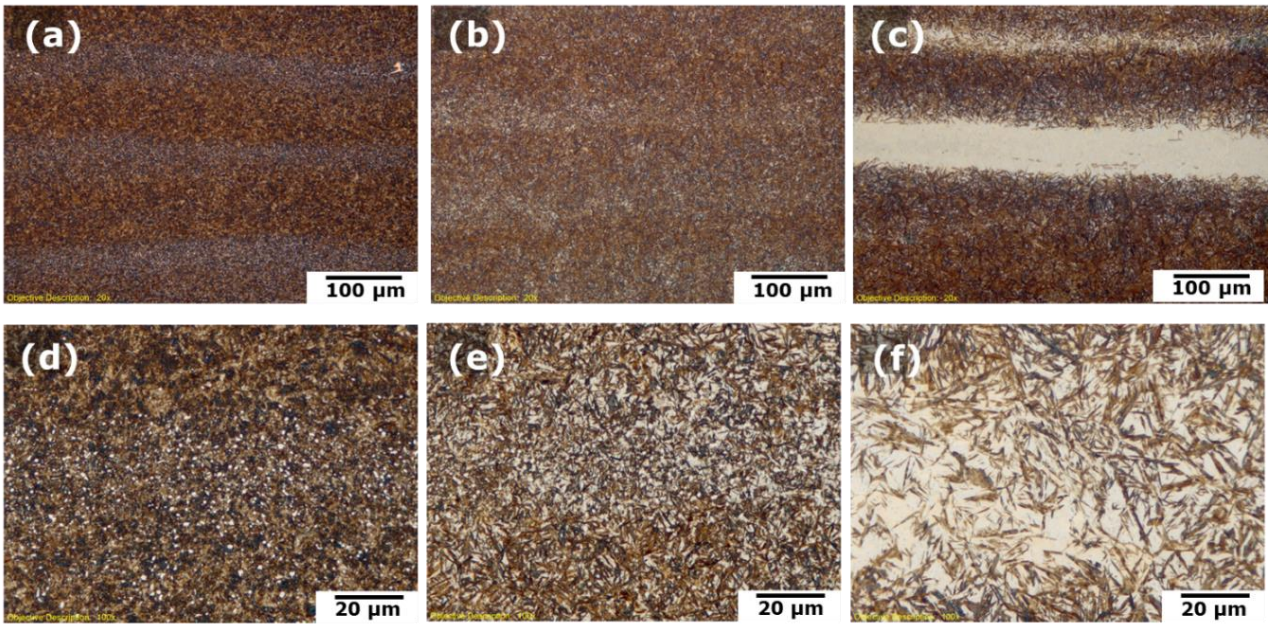


Figure 5. 2 Representative optical micrographs of A485-2 bearing steel showing martensite, retained austenite segregations and carbide segregations at 200x on sample #1(a), sample #2(b) and sample # 3(c); with finer structure indicated in images (d), (e) and (f)

Table 5. 2 Estimation by optical metallographic analysis of the amount of retained austenite (in vol. %) at different depths on induction hardened samples

Sample# / Depth [mm]	Sample #1 (I.H. at 810 °C)			Sample #2 (I.H. at 880 °C)			Sample #3 (I.H. at 980 °C)		
0.05	<5	<5	<5	20.4	20.6	17.2	29.2	26.2	33.6
0.2	9.5	5.6	8.4	24.5	20.3	19.3	26.1	37.1	78.4
0.5	<5	<5	<5	20.1	21.6	22.9	66.8	24.4	34.3
1	<5	<5	<5	21.4	18.7	19.0	32.0	17.1	72.3
1.5	<5	<5	<5	18.6	18.7	18.8	34.3	22.6	43.7
2	<5	<5	<5	14.7	16.8	17.0	24.3	43.7	30.2

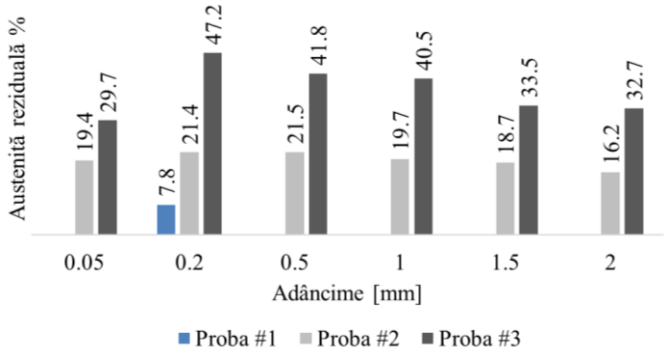
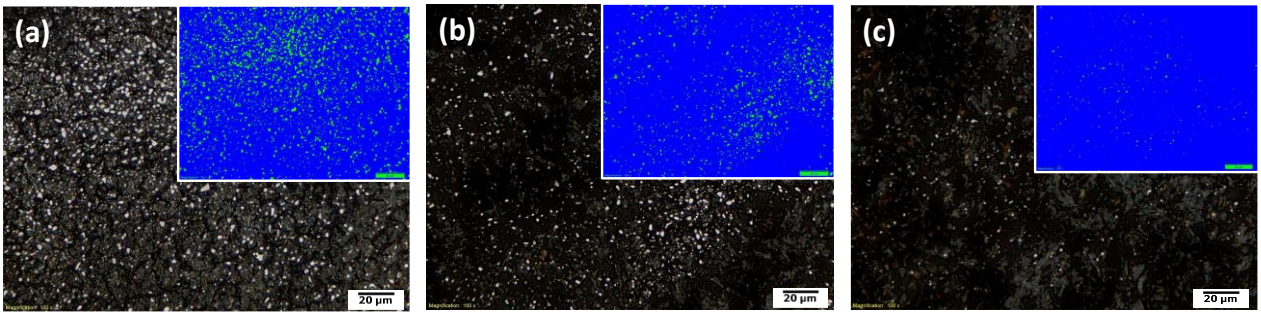


Figure 5. 21 Graphic representation of the average of the results obtained by optical metallographic analysis of the retained austenite content in induction hardened samples (in vol. %) at different depths

The samples show an inhomogeneous microstructure which is described by the martensitic background characteristic of martensitic hardening in steels and segregations of carbides or of retained austenite, or mixed segregations of austenite that also contain carbides. Because of the microstructural inhomogeneity, it was difficult to do a quantitative analysis of the retained austenite because it was necessary to check at least 3 fields at each depth in order to obtain a representative result consisting of the average of the three values. Therefore, table 5.3 shows the estimated values for each sample and the averages of the results obtained at each depth. It should be mentioned that on the sample heat-treated by induction hardening at 810 °C, only carbide segregations are observed at most of the depths at which the evaluations were made, less at the depth of 0.2 mm where a small percentage of retained austenite could be quantified.

After estimating the content of retained austenite through qualitative and quantitative microscopy analysis on each sample, the presence of carbides in the microstructures of the samples was evaluated because in the literature it is mentioned that certain carbides are paramagnetic, something that could influence the results estimated by the magneto-inductive method with Feritscope®. To highlight the carbides on each sample, they were etched with 10% Nital for 3

minutes to obtain a sufficiently good contrast so that the images could be processed using ImageJ software. The percentage of carbides estimated on each sample is shown in figure 5.24.



Proba #1
Temp. călire prin inducție 810 °C

Proba #2
Temp. călire prin inducție 880 °C

Proba #3
Temp. călite prin inducție 980 °C

Figure 5. 23 Detection of carbides and their quantification with the ImageJ software tool in induction-tempered parts at the following temperatures: 810 °C - sample #1 (a); 880 °C - sample #2 (b) and 980 °C - sample #3 (c)

Table 5. 3 Carbide content estimation (vol.%) by diffractometry and optical microscopy

Carbides (vol.%) (Fe, Cr) 3 C	**OM**	**XRD**
Sample #1	14	4.8
Sample #2	8.5	0.98
Sample #3	3	0.78

Table 5.3 presents the comparative results for the estimation of the percentage of carbides, by X-ray diffractometry analysis and by metallographic analysis. Only orthorhombic carbides of the Fe_3C type were measured by X-ray diffraction. In the case of ASTM A485-2 steel where there are alloying elements like Fe, Mo, Cr, Mn which all form carbides, pure carbide (Fe3C) can never be obtained. Thus these carbides were called chromium-enriched carbides, a result confirmed by the images with the distribution of the alloying elements and the local chemical microcomposition by EDS.

5.3 *Results regarding the estimation of retained austenite content by magneto-inductive method*

In the case of inductioned hardened samples, the evaluations performed on the surface of the sample with Feritscope® were carried out without using the correction factor recommended by the equipment manufacturer, and this is due to the fact that the surface of the rectangular bars used is flat. Surface preparation consisted of sanding with fine sandpaper followed by rubbing with ethyl alcohol to leave a clean surface with as fine a roughness as possible. In this situation we consider that the estimated value (AR_m) of retained austenite is the same as the total value (AR_T).

Tabel 5. 4 Retained austenite content (in vol. %) estimated at different depths by magneto-inductive method on induction-hardened samples.

Depth [mm]	Sample #1	Sample #2	Sample #3
0.05	39.5 ± 0.3	38.5 ± 1.4	34.7 ± 0.3
0.2	39.6 ± 0.2	37.9 ± 0.7	34.7 ± 0.9
0.5	37.6 ± 0.9	36.3 ± 1.1	32.9 ± 0.9
1	38.5 ± 0.3	36.2 ± 0.8	33.1 ± 0.4
1.5	38.4 ± 0.2	36.1 ± 0.7	32.8 ± 0.8
2	38.5 ± 0.2	34.6 ± 0.4	32.6 ± 0.4

Thus, the estimated percentages of (RA) at different depths for all samples are presented in Table 5.4. It can be seen that the RA values estimated at different depths are comparable to those obtained by XRD and optical microscopy in the case of sample #3, for samples #1 and #2 the results are much higher than those measured by destructive methods. The total values of the retained austenite (RA_T) estimated by the surface magneto-inductive method are graphically represented alongside the values obtained by X-ray diffraction and image analysis (Figure 5.25 (a), (b) and (c)).

Comparative analysis of the results

To compare the results obtained by means of each analysis method, the measured retained austenite values are graphically represented in Figure 5.25 (a), (b) and (c) for an easier visual comparison.

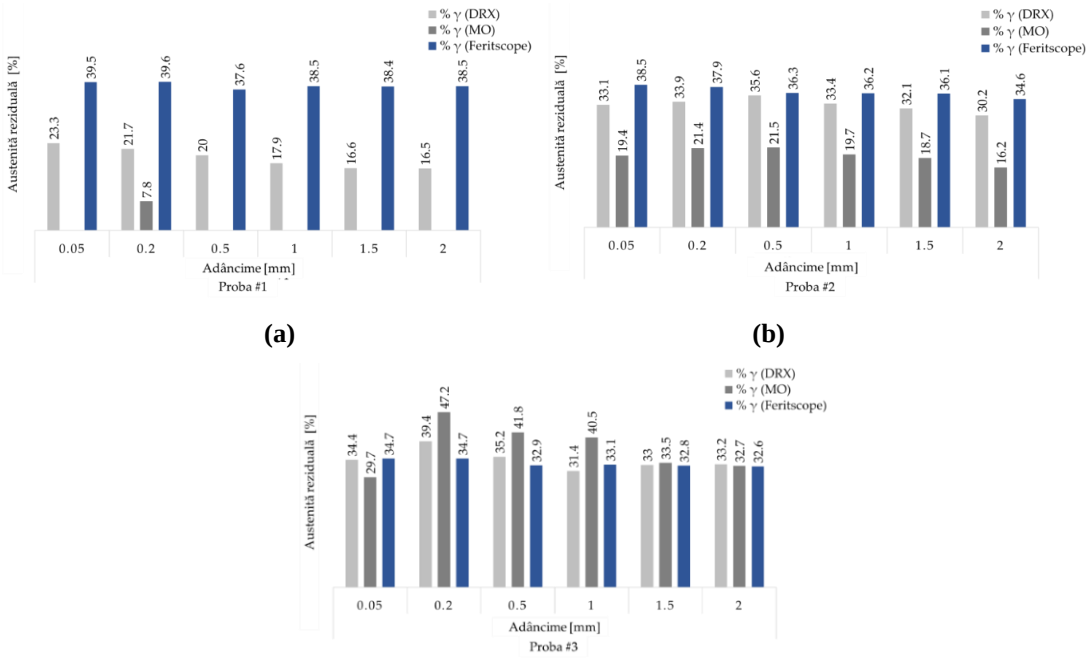


Figure 5. 25 Comparison of AR values estimated by all three methods: Feritscope®, X-ray diffraction (XRD) and quantitative image analysis using optical micrographs on sample #1 (a), Sample #2 (b) and sample #3 (b)

It should be noted that for sample #1, the retained austenite content (estimated on the images processed with the optical microscope) was low and therefore only the value from the depth of 0.2 mm was noted in the graphic representation, at the other depths at which performed investigations could not be evaluated on the microstructure. This indicates to us an inadequate sensitivity of the method in estimating a content of less than 5% RA. Feritscope® is an easy and fast tool to estimate the ferromagnetic content of a steel part. In order to obtain consistent results that are comparable to those obtained by XRD and metallographic image analysis, the measurements were performed in the same area where the samples were analyzed with the other two analysis techniques, XRD and OM.

The following discussion focuses on comparing the techniques in detail. The experimental values obtained are presented in table 5.5, and their average and standard deviation are presented in Table 5.6.

Table 5. 5 Results of retained austenite content estimated by methods: X-ray diffraction (XRD), quantitative image analysis using optical micrographs and Feritscope®

Method	**Depth [mm]**	**I.H. at 810°C (Sample #1)**	**I.H. at 880°C (Sample #2)**	**I.H. at 980°C (Sample #3)**
XRD	0.05	23.3 ± 1.4	33.1 ± 1.4	34.4 ± 3.1
	0.2	21.7 ± 1.5	33.9 ± 1.2	39.4 ± 1.2
	0.5	20.0 ± 0.8	35.6 ± 1.1	35.2 ± 2.5
	1	17.9 ± 0.9	33.4 ± 1.9	31.4 ± 1.7
	1.5	16.6 ± 1.6	32.1 ± 1.2	33.0 ± 2.0
	2	16.5 ± 0.8	30.2 ± 1.5	33.2 ± 0.5
OM	0.05	<5	19.4 ± 1.9	29.7 ± 3.7
	0.2	7.8 ± 2.0	21.4 ± 2.7	47.2 ± 27.6
	0.5	<5	21.5 ± 1.4	41.8 ± 22.2
	1	<5	19.7 ± 1.5	40.5 ± 28.5
	1.5	<5	18.7 ± 0.1	33.5 ± 10.6
	2	<5	16.2 ± 1.3	32.7 ± 10.0
Feritscope®	0.05	39.5 ± 0.3	38.5 ± 1.4	34.7 ± 0.3
	0.2	39.6 ± 0.2	37.9 ± 0.7	34.7 ± 0.9
	0.5	37.6 ± 0.9	36.3 ± 1.1	32.9 ± 0.9
	1	38.5 ± 0.3	36.2 ± 0.8	33.1 ± 0.4
	1.5	38.4 ± 0.2	36.1 ± 0.7	32.8 ± 0.8
	2	38.5 ± 0.2	34.6 ± 0.4	32.6 ± 0.4

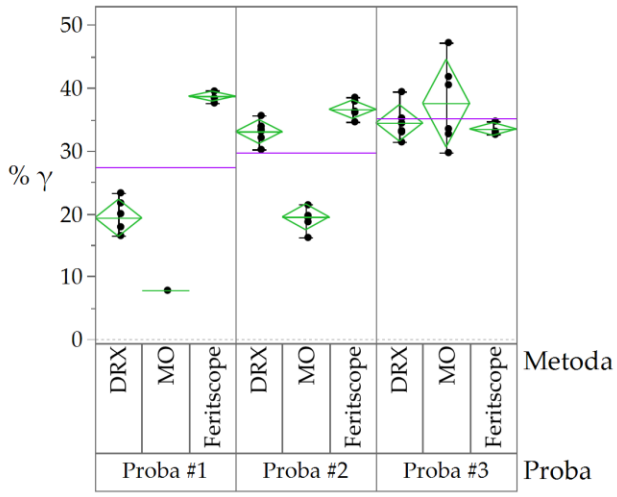


Figure 5. 27 Comparison of retained austenite content estimated with each of the following analysis methods: Feritscope®, optical microscopy and X-ray diffraction

Table 5. 6 Mean values and standard deviations calculated to facilitate the comparison of retained austenite results estimated by the XRD, OM and Feritscope® methods

Samples	Method	Nr.	Mean	Standard Deviation
#1	Feritscope®	6	38.68	0.75
	OM	1	7.8	-
	XRD	6	19.33	2.80
#2	Feritscope®	6	36.60	1.40
	OM	6	19.48	1.96
	XRD	6	33.05	1.81
#3	Feritscope®	6	33.46	0.97
	OM	6	37.57	6.65
	XRD	6	34.43	2.76

Analysis of Variance (ANOVA), is a statistical method that was used to compare the mean values of retained austenite in A485-2 steel estimated by Feritscope® compared to X-ray diffraction (XRD) and optical microscopy (OM). Depending on the percentage of phases in the microstructure of the samples, the three analysis methods provided representative results for the characteristics of each sample. The final average of the values consists of the average of the 6 evaluations obtained at different depths at which the determinations were made, namely: surface, 0.2 mm, 0.5 mm, 1 mm, 1.5 mm and 2 mm.

The test area for both XRD and OM can be considered almost the same. This is due to the fact that the results of the metallographic analysis are evaluated on the surface of the sample, and

X-rays penetrate a few micrometers, thus the methods are limited to the subsurface area. The values shown in Table 5.6 were calculated as percentage differences in AR content between the different methods used for the current study. The percentage difference factor was used in particular to provide a comparison factor other than the difference between the absolute values of the retained austenite content.

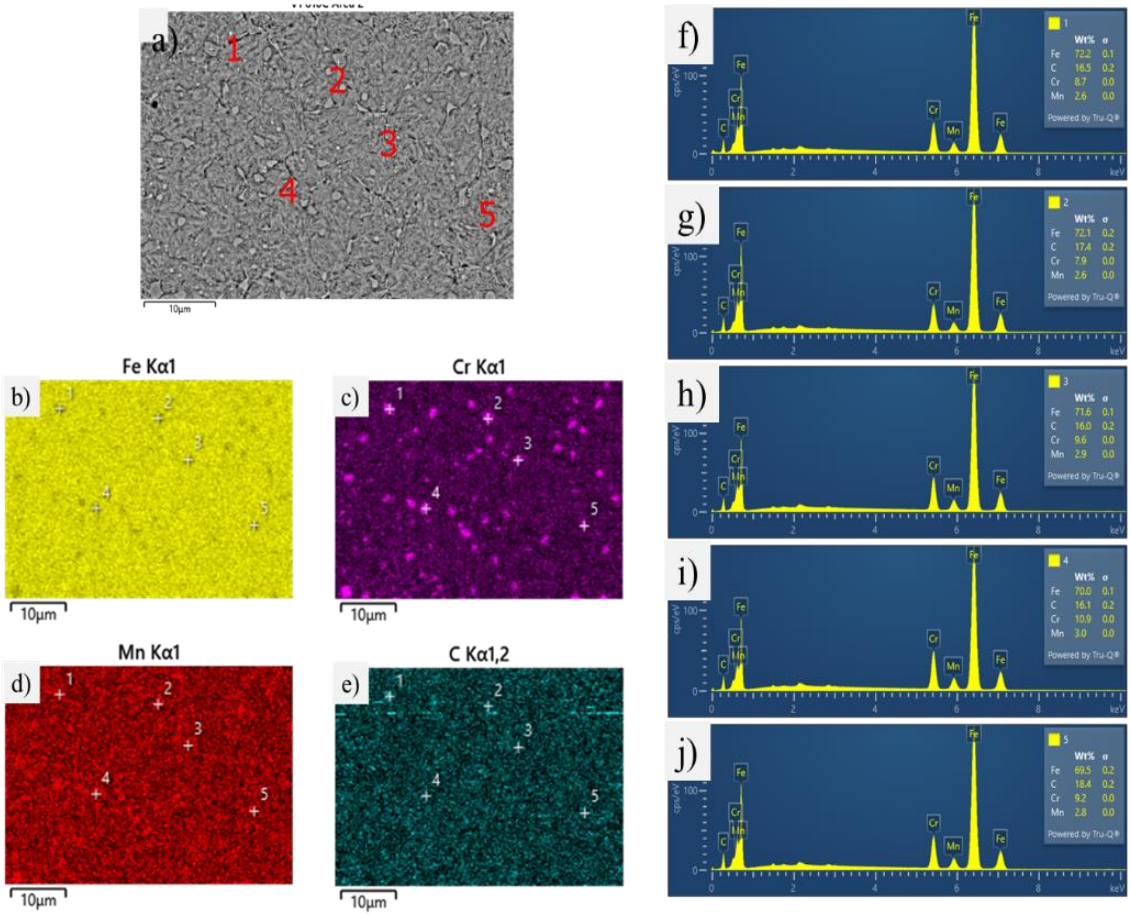
5.4 Results regarding the analysis with the scanning electron microscope

Scanning electron microscope analysis of the induction hardened samples, shown in Fig. 5.29- Fig. 5.32, comes to complete the information on the morphology of the samples surfaces .

Scanning electron microscope analysis of experimental samples provides information on the nature of the carbides precipitated in the material, as well as the distribution of alloying elements.

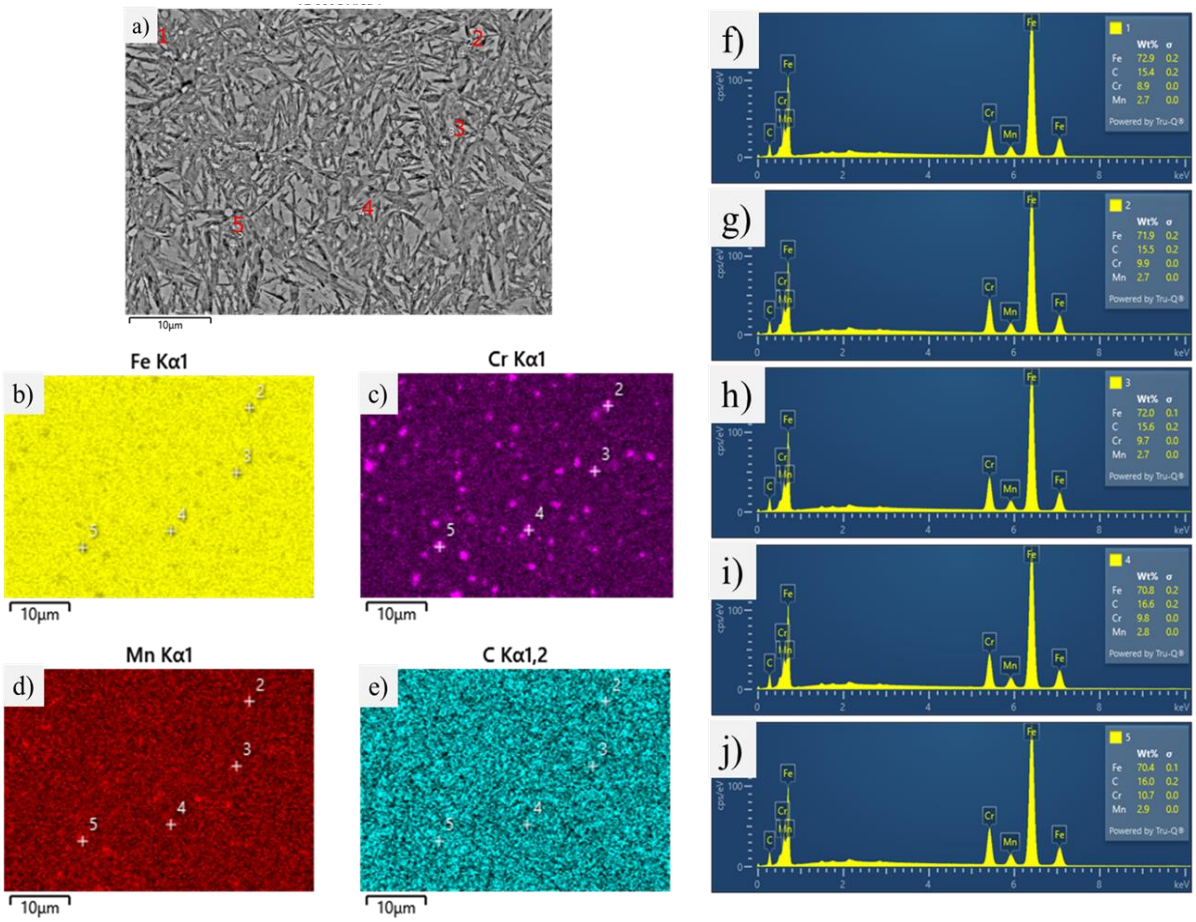
Following the optical and electronic microscope analyses, it was noted that in induction hardened samples, the volume content of carbides is significant enough to influence the results obtained using Feritscope®, therefore they can affect the estimated content of retained austenite. Indeed, although the iron carbide (cementite) which is ferromagnetic is found in a fairly large percentage on the microstructure of the induction hardened samples, the rest of the carbides formed in the steel could be of a paramagnetic nature, such as chromium carbide and depending on the percentage in which they are found can distort the final result of the magneto-inductive method [99], [100]. The quality of steel considered in the present study has a sufficient content of Cr and Mn to form a significant amount of carbides affecting the result of the evaluations carried out by the magneto-inductive method, and the fraction of Cr or Mn carbides is expected to be higher than the other types of carbides. Since the majority of the carbide fraction in the A485-2 steel grade is that of chromium carbides, it is likely to make a sum with the paramagnetic response of retained austenite provided by Feritscope®. As a result, the percentage of retained austenite indicated by Feritscope® is expected to be higher than the actual content [7], [99], [101].

In conclusion, estimates by the magneto-inductive method using a Feritscope® overestimate the amount of retained austenite compared to X-ray diffraction (XRD) and optical microscope (OM) image processing.



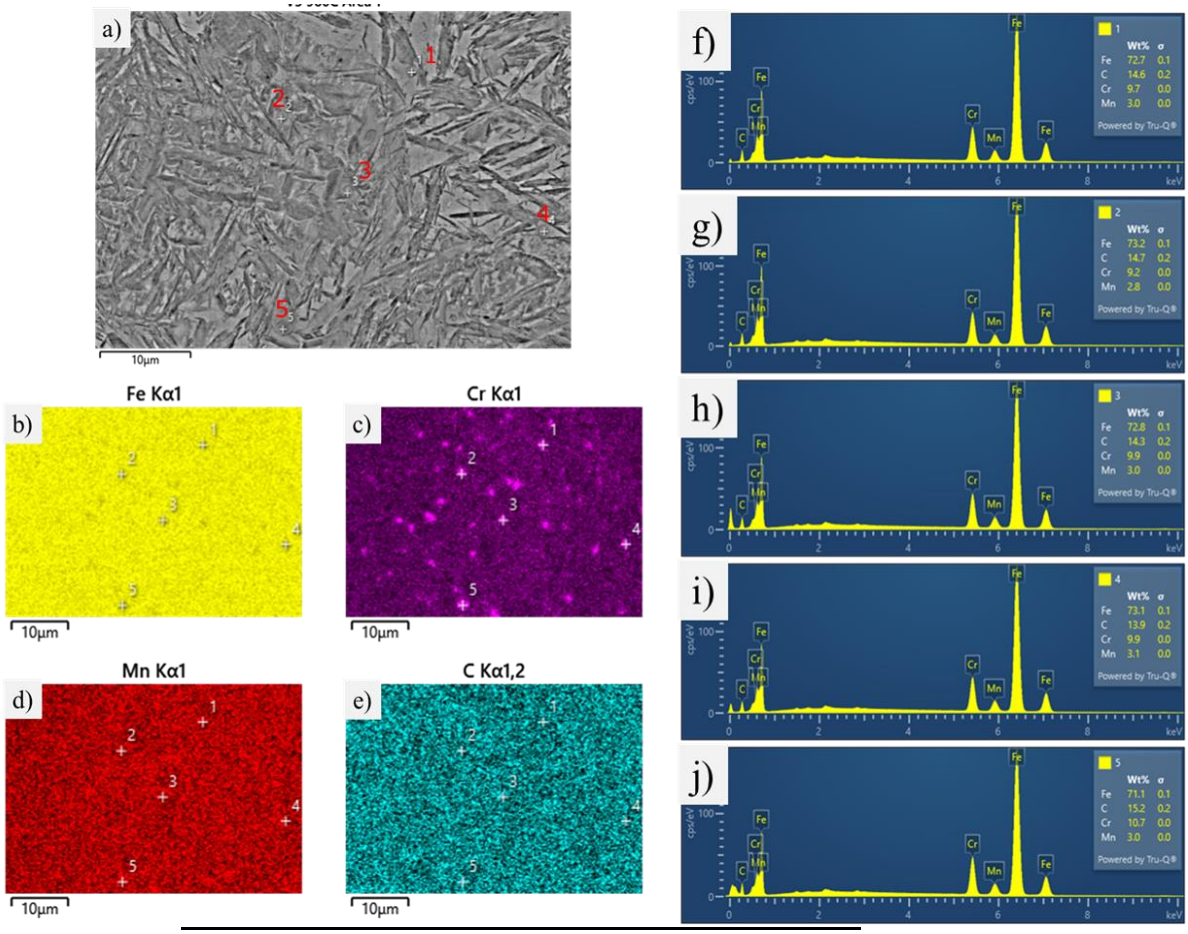
Element	Micro-compoziţia locală, wt[%]			
	C	Cr	Mn	Fe
1	16.74	8.66	2.64	72.23
2	17.45	7.89	2.55	72.12
3	15.98	9.55	2.88	71.58
4	16.08	10.95	3.02	69.96
5	18.43	9.24	2.8	69.53

Figure 5. 29 Scanning electron microscope analysis of the sample induction hardened at 810°C: (a) - SEM image, (b), (c), (d), (e) - Secondary electron images with the distribution of the elements alloy; (f) – (j) - EDS on the fields indicated in image (a); local microcomposition



Element	Micro-compoziţia locală, wt[%]			
	C	Cr	Mn	Fe
1	15.45	8.95	2.68	72.93
2	15.46	9.95	2.70	71.90
3	15.59	9.66	2.71	72.05
4	16.64	9.78	2.76	70.82
5	16.02	10.68	2.89	70.42

Figure 5. 30 Scanning electron microscope analysis of the surface hardened sample at 880°C: (a) - SEM image, (b), (c), (d), (e) - Secondary electron images with the distribution of the elements alloy; (f) – (j) - EDS on the fields indicated in image (a); local microcomposition



Element	Micro-compoziţia locală, wt[%]			
	C	Cr	Mn	Fe
1	14.58	9.68	3.01	72.72
2	14.73	9.25	2.81	73.21
3	14.30	9.92	3.01	72.77
4	13.92	9.93	3.05	73.09
5	15.20	10.71	3.01	71.08

Figure 5. 31 Scanning electron microscope analysis of the surface hardened sample at 980°C: (a) - SEM image, (b), (c), (d), (e) - Secondary electron images with the distribution of the elements of alloy; (f) – (j) - EDS on the fields indicated in image (a); local microcomposition

CONCLUSIONS. ORIGINAL CONTRIBUTIONS. PERSPECTIVES FOR FUTURE RESEARCH

The experimental research carried out in the structure of this doctoral thesis was directed towards the fulfillment of the originally proposed objective. The objective of the paper is to study the magneto-inductive method used to evaluate different levels of retained austenite in samples prepared from case-hardening steel brand A534B29, as well as from surface-hardened steel brand

A485-2, comparing the level measurements over time. X-ray diffractometry and qualitative and quantitative metallographic analysis of the microstructures obtained under the optical microscope. Compared to the original microstructure, the retained austenite content in the A534B29 case-hardening steel was transformed by additional tempering treatment, thus obtaining three samples with distinct ranges of retained austenite content in the carburized case depth of the steel part. In the case of A485-2 type bearing steel, the samples were induction hardened at different temperatures, subsequently performing the cryogenic treatment and tempering to the same temperatures in the case of each hardened sample.

A. Conclusions derived from documentary research

For the assessment of retained austenite content in different steels, the quantitative and qualitative optical metallographic analysis (OM), the magneto-inductive method analysis (Feritscope®), the X-ray diffraction analysis (XRD) were studied as reference methods, completing the analysis in the scanning electron microscope. While OM and XRD have been considered reference methods for the validation of the Feritscope® result, it is worth noting the factors that could affect the accuracy and efficiency of these techniques. OM with image contrast counting techniques can produce reliable results of phase content. However, the need for the destructive preparation of several embedded samples while following a long metallographic process and the quantification of several regions makes this technique extremely time-inefficient. Quantification of retained austenite and martensite phases with this technique is often difficult due to the very small size of the carbides in the microstructure. Although the carbide fraction is considerably lower, it adds to the retained austenite (RA) fraction because the carbides also appear bright in the microstructure (using nital etching), thus affecting the RA content value estimated by MO. In addition, since the evaluation is limited to the surface fraction by OM, anisotropically grown grains could give results that deviate from the actual content.

Quantitative X-ray diffraction (XRD) techniques are considered more accurate techniques than quantitative and qualitative metallography analysis for the quantification of retained austenite. The results are more reliable with XRD, as it can overcome the resolution difficulties of optical microscopy. Standard XRD techniques (such as the internal standard and peak area surface) have limitations for complex multiphase materials. Although carbide correction can be done, it is very difficult to measure carbide fractions with other techniques. Additional issues affecting accuracy include preferential orientation and peaks representative of overlapping carbides. The X-ray diffraction (XRD) technique can be considered quasi-destructive when sample preparation is required for deep retained austenite profiling and during the evaluation of complex shaped components. In addition, both X-ray diffraction and optical microscopy require trained personnel to operate them.

The Feritscope® is relatively easy to operate with fast efficiency and any complex shaped steel component with a finished surface can be tested. Careful considerations are required when testing the component, such as maintaining the required distance between the probe and the sample and considering the correction factor for curved surfaces to obtain results closer to the actual

content. While if the carbide fraction is considered sufficiently insignificant it does not affect the results, there is also the case where it can still affect the results when its fraction is comparable to that of the retained austenite. Interestingly, several studies have reported that some iron carbides are ferromagnetic. Since the majority of the carbide fraction in steel is iron carbide, it is likely to bond with the ferromagnetic response of martensite provided by Feritscope®. As a result, the RA content estimated using Feritscope® is likely to be closer to the actual content, avoiding interference with carbides. However, it is worth noting that carbides other than iron carbides, such as chromium carbides, could be paramagnetic and interfere with the final result of the magneto-inductive method. However, the quality of the steel considered in the present study has a sufficiently low Cr and Mo content to not form a significant amount of carbide that could affect the result obtained with the magneto-inductive method, and the iron carbide fraction is expected to be greater than the rest of the carbide proportion.

B. Conclusions derived from own experimental research

B. 1 Conclusions regarding the estimation of the amount of retained austenite in bearing steels heat treated by carburizing:

The results regarding the estimation of retained austenite (RA) content evaluated at different depths are comparable, both with the X-ray diffraction (XRD) method and with the optical metallographic method. At the same time, the insignificant deviation of the values of the RA contents estimated by Feritscope® indicates a precise measurement of the retained austenite compared to the XRD. It can be concluded that the measurement method with Feritscope® is sensitive enough to detect RA below 5% with values that can be compared to those obtained by X-ray diffraction (XRD).

The results regarding the estimation of retained austenite content in carburized bearing steels evaluated by the magneto-inductive method, are comparable to the results obtained by XRD and MO and led to the following conclusion:

- The results of Feritscope® estimations on the sample surfaces revealed a direct correlation with both destructive methods such as XRD and MO;
- The average percentage difference of the results estimated by the Feritscope®, XRD, and MO % analysis methods for the samples with low and medium retained austenite (RA) content was in the range of 5-31%;
- The average percent difference in retained austenite (RA) estimated by Feritscope® compared to XRD and MO increased as the actual retained austenite (RA) content decreased, while the absolute difference between the estimated RA values remained consistent;
- Feritscope® detected a content of retained austenite (RA) below 5% and the results provided were comparable to the values measured by XRD and MO;
- Comparative evaluation of Feritscope® with the widely used XRD and OM techniques, it was observed that Feritscope®, with faster detection and less time consumption, provides

comparable results in the case of different levels of RA sufficient to satisfactory for industrial purposes

Conclusions on estimating the amount of retained austenite in bearing steels heat treated by induction hardening:

The results regarding the estimation of the proportion of retained austenite in bearing steels obtained by induction using X-ray diffraction showed that the proportion of retained austenite is dependent on the temperature at which the experimental samples were induction hardened. Thus:

- The average RA content of the induction hardened sample at 810°C decreased from 23.3% to 20.0% up to 0.5 mm, with a constant decreasing trend to the value of 16.5% at a depth of 2 mm. On the other hand, in the samples that were treated at a higher temperature (samples #2 and #3), the proportion of general RA decreased by a lower percentage compared to sample #1.
- The average RA content of the induction-hardened sample at 880°C steadily decreases from 33.1% at the surface to 30.2% at 2 mm, with a percentage value of 9%.
- The average RA content of the induction-hardened sample at 980°C has no trend concerning the proportion of RA. The RA values seem to fluctuate from the surface up to 2 mm in a range of 39.4% to 31.4% and this is because segregations of retained austenite are present. However, a slight decrease in the average retained austenite values from the surface to a depth of 2 mm can be observed on each sample.

The results regarding the estimation of the proportion of retained austenite in bearing steels obtained by induction hardening using the optical metallographic method highlighted the following conclusions:

- During the surface analysis of the experimental samples at the temperature of 810 °C, only segregations of carbides are identified, with the increase of the surface tempering temperature by induction to 880 °C, along with the carbides, segregations of retained austenite also appear, because at the temperature of 980 °C Only bands of retained austenite appear;
- At a depth of 0.2mm, after surface quenching at 810°C, only carbide segregations and a small amount of retained austenite also appear. When quenching at 880 °C, segregations of retained austenite mixed with carbides appear, so that at 980°C only segregations of retained austenite can be distinguished;
- At a depth of 0.5 mm, after surface tempering at 810 °C, only carbide segregations also appear. In the case of the sample treated at 880°C, only segregations of retained austenite and a reduced amount of carbides appear, and at 980°C only segregations of retained austenite can also be distinguished;
- At a depth of 1 mm, the proportion of carbides begins to decrease in all situations, respectively, at surface tempering at 810°C, carbide segregations are substantially reduced, they appear sporadically at a surface tempering at 880 °C, and at 980 °C distinguish only retained austenite segregations;

- When the depth increases to 1.5 mm and 2 mm, the aspects are similar in the case of the sample treated at low temperature, the segregations of carbides are reduced, the microstructure becomes more homogeneous, and in the case of samples #2 and #3 sporadic segregations of retained austenite and carbides are recorded;
- By using image analysis software, it was also possible to quantify the percentage volume content of carbides present on the microstructure of the induction-hardened samples, observing its decrease as the surface induction-hardening temperature increased. Thus the proportion of carbides decreases from a value of 14% at a tempering temperature of 810°C, to a value of 8.5% at a tempering temperature of 880°C, reaching 3% carbides at a temperature of 980°C.

The results regarding the estimation of the proportion of retained austenite in the standard steels obtained by magneto-inductive analysis (Feritscope®) highlighted the following conclusions:

- The values of the retained austenite content (RA) estimated at different depths are comparable to those obtained by X-ray diffraction (XRD) and optical microscopy for the case of the induction hardened sample at 980°C;
- The values of the proportion of retained austenite for the samples tempered superficially at 810° and 880°C are higher compared to the results obtained by the other destructive methods;
- By using the ANOVA statistical method, the results regarding the retained austenite content estimated by each method, respectively Feritscope® compared to X-ray diffraction (XRD) and OM, could be compared. Depending on the percentage of phases in the microstructure of each sample, the three analysis methods provided representative results for the characteristics of each sample. The final average of the values consists of the average of the 6 evaluations obtained at the different analysis depths, namely: surface, 0.2 mm, 0.5 mm, 1 mm, 1.5 mm, and 2 mm. The highest standard deviation value was found to be given by the results estimated by metallographic analysis.

The results regarding the estimation of the proportion of retained austenite in bearing steels obtained by scanning electron microscope analysis led to the following conclusions:

- In the sample hardened superficially at 810°C, in the five fields on the surface of the sample, complex carbides of chromium and manganese could be identified, with a relatively homogeneous distribution of the alloying elements. The chromium content is in the range of 7.89%-10.95%, and the manganese in the range of 2.55%-3.02%. Noteworthy is the fine structure of the martensite;
- In the sample hardened superficially at 880°C, complex carbides of chromium and manganese could also be identified in the five fields on the surface of the sample, with a relatively homogeneous distribution of the alloying elements, and the chromium content is located in the range of 8, 95%-10.68%, and that of manganese in the range 2.68%-2.89%. In this situation, the martensite is acicular, with the size of the needles larger than in the case of the sample tempered at 810°C;

- In the sample hardened superficially at 980°C, complex carbides of chromium and manganese could also be identified in the five fields on the surface of the sample, with a relatively homogeneous distribution of the alloying elements, and the chromium content is located in the range of 9, 25%-11.22%, and that of manganese in the range of 2.81%-3.74%. On the microstructure of this sample, martensite shows the largest size of martensitic needles. It can be appreciated that by increasing the surface tempering temperature by induction, the average content of chromium complex carbides increases from 9.258%Cr in the sample tempered at 810°C, to 9.804%Cr in the sample tempered at 880°C, up to 10.205%Cr to the sample tempered at 980°C. The average manganese content of the complex carbides varies between 2.778% (sample at 810°C), 2.748% (for the sample tempered at 880°C), and 3.270%Mn (for the sample tempered at 980°C), respectively.

- The grade of steel considered in the present study has sufficient Cr and Mo content to form a significant amount of carbides to affect the result of measurements obtained by the magneto-inductive method, and the fraction of Cr, Mo, or Mn carbides is expected to be higher than the other types of carbides. Since the major carbide fraction in the A485-2 steel grade is chromium carbide, it is likely to couple the paramagnetic response of the retained austenite to the secondary coil of the Feritscope®. As a result, the RA content estimated by Feritscope® is expected to be higher than the actual content.

ORIGINAL CONTRIBUTIONS

By carrying out the experimental research within the present doctoral thesis, the following original contributions could be made:

- The comparative consideration of two categories of bearing steels for the identification of the retained austenite content and the application of their specific heat treatments, either carburizing or induction hardening, a contribution with great industrial applicability;

- Comparative use of various conventional or non-conventional methods for the determination of retained austenite content, either by destructive methods (X-ray diffraction analysis, metallographic microscope analysis, or scanning electron microscope analysis) or non-destructive methods (magneto-inductive analysis) ;

- The use of the ANOVA statistical method to compare the results regarding the retained austenite content either by non-destructive or destructive methods, respectively the method using Feritscope®, the X-ray diffraction method, the quantitative and qualitative optical metallographic analysis method, the magneto-inductive method and scanning electron microscope analysis method.

PERSPECTIVES OF FUTURE RESEARCH

The possibilities of estimating the amount of retained austenite by non-conventional methods in bearing steels continue to raise a major interest in the industry, therefore numerous directions of further experimental research can be developed:

- Expanding the scope of use of Feritscope®, not only for estimating the amount of delta ferrite in stainless steels but also in the field of metallic materials with structural constituents with paramagnetic properties, such as bearing steels;
- The use of the method of estimating the proportion of retained austenite with the aid of the Feritscope® magneto-inductive method in bearing steels subjected to heat treatment by induction hardening with reduced Cr and Mn content, to check whether there is closeness to the estimation of the actual retained austenite content;
- The use of different comparative methods for determining the proportion of retained austenite for other ranges of bearing steels, other than those studied in this PhD thesis;
- Applying the surface induction hardening heat treatment at other temperatures than those considered in this Ph.D. thesis, for example, 715°C, 725°C, and 800°C, at which other categories of carbides can precipitate in the martensitic matrix, with different proportions of retained austenite.

SELECTIVE BIBLIOGRAPHY

[1] H. Fu and P. E. J. Rivera-Díaz-del-Castillo, "A unified theory for microstructural alterations in bearing steels under rolling contact fatigue," *Acta Mater*, vol. 155, pp. 43–55, Aug. 2018, doi: 10.1016/j.actamat.2018.05.056.

[4] A. Warhadpande, F. Sadeghi, and R. D. Evans, "Microstructural alterations in bearing steels under rolling contact fatigue part 1-Historical overview," *Tribology Transactions*, vol. 56, no. 3, pp. 349–358, 2013, doi: 10.1080/10402004.2012.754073.

[7] S. Talebi, M. Jahazi, and H. Melkonyan, "Retained Austenite Decomposition and Carbide Precipitation during Isothermal Tempering of a Medium-Carbon Low-Alloy Bainitic Steel," *Materials*, vol. 11, no. 8, p. 1441, Aug. 2018, doi: 10.3390/ma11081441.

[20] "Standard Practice for X-Ray Determination of Retained Austenite in Steel with Near Random Crystallographic Orientation 1," *ASTM International*, 2019, doi: 10.1520/E0975-13.

[24] C. F. Jatczak, "Retained Austenite and Its Measurement by X-Ray Diffraction," Feb. 1980. doi: 10.4271/800426.

[25] J Taptich, M Brauss, M Belassel, G Grodzicki, T Simmons, J Nantais "Characterization of Residual Stresses and Retained Austenite on 416 Stainless Steel via X-Ray Diffraction Techniques," Oct. 2018, pp. 159–164. doi: 10.21741/9781945291890-25.

[26] V. Lejay, C. Sidoroff, C. Le Bourlot, M. Perez, and P. Dierickx, "Ageing of 100Cr6 steel: synchrotron X-ray diffraction and dimensional analysis investigation," *Materials Science and Technology*, vol. 32, no. 11, pp. 1106–1110, Jul. 2016, doi: 10.1080/02670836.2015.1136102.

[27] E. Jimenez-Melero, R. Blondé, M. Y. Sherif, V. Honkimäki, and N. H. van Dijk, "Time-dependent synchrotron X-ray diffraction on the austenite decomposition kinetics in SAE 52100 bearing steel at elevated temperatures under tensile stress," *Acta Mater*, vol. 61, no. 4, pp. 1154–1166, Feb. 2013, doi: 10.1016/j.actamat.2012.10.025.

[28] George Krauss, "Carburizing: Processing and Performance : Proceedings of an International Conference ," George Krauss, Ed., ASM International, 1989, Jul. 1989.

[29] G. Krauss, *Steels: Heat Treatment and Processing Principles*, Second Edition. ASM International.

[31] S. H. Magner, R. J. De Angelis, W. N. Weins, and J. D. Makinson, "A HISTORICAL REVIEW OF RETAINED AUSTENITE AND ITS MEASUREMENT BY X-RAY DIFFRACTION."

[33] L. G. Ionescu, M. V. Pantawane, C. Tănase, R. V. Sichim, C. A. Dascălu, and B. Ghiban, "Evaluation of Retained Austenite in Carburized Bearing Steel Using Magneto-Inductive Method," *Crystals (Basel)*, vol. 13, no. 8, p. 1173, Jul. 2023, doi: 10.3390/cryst13081173.

[34] R. Kshirsagar, S. Jones, J. Lawrence, and J. Tabor, "Measurement of ferrite content of stainless steel sheet welds using a new Ferrite Density Number scale," *J Mater Process Technol*, vol. 274, Dec. 2019, doi: 10.1016/j.jmatprotec.2019.116278.

[35] J. Arnold and T. Gnäupel-Herold, "A comparison of different measurement methods to evaluate strain-induced martensite formation in a metastable austenitic stainless steel," 2014. [Online]. Available: https://www.researchgate.net/publication/289576653

[65] G. F. Vander Voort, "Image Analysis," in *Materials Characterization*, ASM International, 1986, pp. 309–322. doi: 10.31399/asm.hb.v10.a0001755.

[66] S. Su *et al.*, "A Fe-Cr-C steel based on gradient scale precipitation hardening: Hardening and toughening mechanism of multistage heat treatment," *J Alloys Compd*, vol. 946, p. 169355, Jun. 2023, doi: 10.1016/j.jallcom.2023.169355.

[67] A. D. Anoop, A. S. Sekhar, M. Kamaraj, and K. Gopinath, "Numerical evaluation of subsurface stress field under elastohydrodynamic line contact for AISI 52100 bearing steel with retained austenite," *Wear*, vol. 330–331, pp. 636–642, May 2015, doi: 10.1016/j.wear.2015.01.021.

[68] R. L. Banerjee, "X-ray determination of retained austenite," *Journal of Heat Treating*, vol. 2, no. 2, pp. 147–150, Dec. 1981, doi: 10.1007/BF02833231.

[71] *Standard Specification for High Hardenability Antifriction Bearing Steel*. USA: ASTM International, 2022. doi: 10.1520/A0485-17R22.

[90] E. Claesson, H. Magnusson, J. Kohlbrecher, M. Thuvander, and P. Hedström, "Evolution of iron carbides during tempering of low-alloy tool steel studied with polarized small angle neutron scattering, electron microscopy and atom probe," *Mater Charact*, vol. 194, Dec. 2022, doi: 10.1016/j.matchar.2022.112464.

[100] J. Suwalski, Z. Kucharski, M. Łukasiak, and W. Luty, "Determination of retained austenite in bearing steel," *Hyperfine Interact*, vol. 29, no. 1–4, pp. 1491–1494, Feb. 1986, doi: 10.1007/BF02399517.

[101] D. W. Hetzner and W. Van Geertruyden, "Crystallography and metallography of carbides in high alloy steels," *Mater Charact*, vol. 59, no. 7, pp. 825–841, Jul. 2008, doi: 10.1016/j.matchar.2007.07.005

LIST OF PUBLICATIONS FOR DISSEMINATION OF RESULTS

Articles published in ISI&Web of Science rated journals

1. L. G. Ionescu, C. Tanase, M. Vasilescu, R. Zamfir, and B. Ghiban, "Research related to retained austenite on rail and windmill bearings" Bull., Series B, vol. 83, no. 2, p. 2021.

2. L. G. Ionescu, M. V. Pantawane, C. Tănase, R. V. Sichim, C. A. Dascălu, and B. Ghiban, "Evaluation of Retained Austenite in Carburized Bearing Steel Using Magneto-Inductive Method" Crystals (Basel), vol. 13, no. 8, p. 1173, Jul. 2023, doi: 10.3390/cryst13081173

Articles presented at international conferences

Laura G. IONESCU, Brânduşa GHIBAN- Nondestructive techniques for retained austenite measurement, 9 th International Conference on Materials Science and Technologies – RoMAT 2022 November 24-25, 2022, Bucharest, Romania www.romat2022.ro