# CORRELATION BETWEEN NDT MEASUREMENTS AND SIGMA PHASE CONTENTS IN DUPLEX STAINLESS STEELS

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**ABSTRACT.** Direct measurements of sigma phase content with electron microscopy and X-Ray diffraction methods are impractical. Eddy current (conventional sorting procedure and algorithm for assessment of electrical conductivity and magnetic permeability) and induced positron analysis were investigated as alternative quantitative techniques. Direct X-Ray diffraction measurements were also conducted. Nondestructive testing indications were compared to metallographic tests performed on twin specimens. Eddy current techniques proved to be very sensitive to phase transformations in duplex stainless steels.

**Keywords:** Eddy Current Testing, Advanced Eddy Current Techniques, Magnetic Permeability, Electrical Conductivity, Duplex Stainless Steel, Sigma Phase, Metal Phase Transformations, X-ray Diffraction, Induced Positron Analysis.
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## INTRODUCTION

The presence of sigma phase (SP) can be detrimental to the material properties, which explains the stringent quality-control requirements. The ferrite phase (FP) can transform into SP and austenite in duplex stainless steels due to improper heat treatment [1]. In general, the increase of SP content is associated with the decrease of FP content. The FP is magnetic. The sigma and austenite phases are nonmagnetic.

Various destructive and very few nondestructive testing (NDT) methods have been discussed in the literature for detection and measurement of SP [1]. Only metallographic and X-Ray diffraction methods provide direct measurements of SP content [1]. Both methods require time for processing and X-Ray diffraction measurements are limited to the material surface. The use of magnetic and magneto-inductive NDT [1,2] has been considered for FP measurement. On one hand, the absolute level of FP content in any duplex stainless steel material cannot be used directly to measure the SP content because the FP shape and distribution changes for different steel grades and parts and depends on too many factors [1,2]. On the other hand, too little ferrite might be indicative of the formation of intermetallics [1].

Four major NDT techniques were investigated in the present study: conventional eddy current sorting, induced positron analysis, X-Ray diffraction, and advanced eddy current

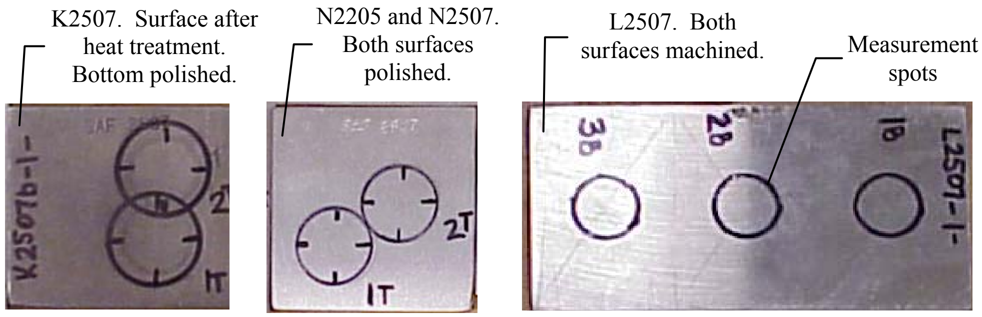


**FIGURE 1.** Specimen groups with measurement spots.

technique for assessment of electrical conductivity (σ) and relative initial magnetic permeability ($\mu_{ri}$).

## EXPERIMENTAL SPECIMENS

A group of 22 duplex stainless steel specimens was tested (Figure 1). Subgroup N2205 specimens were made from steel SAF 2205. Specimens in groups K2507, L2507 and N2507 were made from steel SAF 2507. Datasheets describing the grades are available on the Web (http://www.smt.sandvik.com/tube).

L2507 and K2507 had a thickness of 4.3 and 4.4 mm, respectively. The thickness of N2507 and N2205 was 2.6 mm. Top and bottom surfaces of L2507 had been machined after heat treatment. The top and bottom surfaces of N2507 and N2205 had been machined and polished. Only the bottom surface of K2507 had been machined and polished. The top surface of K2507 had not been mechanically treated (Figure 1) after heat treatment. Three spots uniformly distributed on the surface were specified for each of the L2507 specimens for a total of 21 measurement spots. Two spots per specimen, or a total of 30 measurement spots, were selected for the three groups (K2507, N2507, and N2205) of 15 specimens. The total number of measurement spots was 51.

The SP had been measured destructively by averaging the content throughout the cross section on twin specimens. The ferrite content of specimens K2507, N2507, and N2205 had been measured nondestructively with electromagnetic ferrite meter instrumentation and procedure. Only SP content was known for the group L2507.

The specimen matrix allowed identifying the machining effect comparing the top and bottom surface indications for K2507. The thickness effect could possibly be identified by comparison of indications between K2507 and N2507 for NDT such as volumetric-induced positron analysis. Machining and thickness were not expected to affect the indications for groups N2507 and N2205.

## NDT TECHNIQUES

Techniques for electrical conductivity measurements with eddy current instruments are extensively used in many industries, and aerospace, in particular, for nondestructive characterization of conductive nonmagnetic material hardness, chemical composition, cold working, metallurgical phase transformations, and others [3,4]. The task of magnetic and conductive material characterization is complicated because more electromagnetic parameters are affected. Specimens with a simple shape allow the application of fairly straightforward and fast algorithms for multiple properties measurements [5].

The advanced eddy current technique (AECT) for material property measurement was used during the work on several EWI projects. It consisted of modeling of eddy current probe

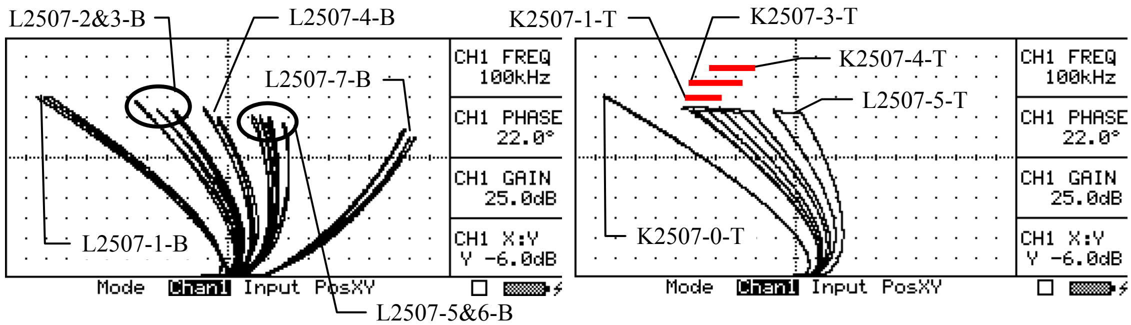


**FIGURE 2.** Typical conventional eddy current sorting at 100 kHz.

response interaction with ferromagnetic steel material. The model was then optimized to match the probe-measured parameters when the probe was positioned on the actual specimens.

The positron-induced photon annihilation technique (http://www.positronsystems.com/) has recently evolved into two separate techniques: induced positron analysis – volumetric (IPA-V) and induced positron analysis – surface (IPA-S). Both techniques have been used in this study.

The X-Ray diffraction is a standard method for metallographic phase content and stress measurements needed for material characterization. A description of the method can be found elsewhere (http://www.gnr.it/xrd.htm).

## Conventional Eddy Current Sorting

The conventional eddy current sorting procedure was applied to compare the results between and within the groups (Figure 2) and to verify the uniformity of material across the specimen surface. The sorting procedure was conducted at four frequencies: 25, 50, 100, and 200 kHz.

The SP content varied from 0.06% (L2507-1) to 10.2% (L2507-7) and from 0.0% (K2507-0) to 5.2% (K2507-5). Further, the FP content varied from 39% (K2507-0) to 23% (K2507-5). The indications obtained on the specimens with the least amount of SP had the least scatter compared to ones obtained on specimens with higher SP. The distance on the screen between the first and the second spot marked on the specimen (Figure 1) for K2507 was highlighted for more clarity with the bars (Figure 2, right) indicating higher scatter leading to overlapping and difficult separation.

In summary, the conventional eddy current sorting techniques were capable of reliably and consistently separating the specimens that did not have SP from those that had it. A well-defined correlation between eddy current indications and SP/FP content was demonstrated. It was not clear, though, what the contribution of each phase on the signal pattern was. It was also determined that the surface machining condition (e.g., machined vs. not machined) affected the sorting results.

## Advanced Eddy Current

The AECT for assessment of $\sigma$ and $\mu_{ri}$ through model optimization involved the following activities: characterization of an existing probe through precision measurements, modeling of the probe interaction with the inspected material, validation of the model, and assessment of unknown material properties through model optimization.

A set of validation measurements was conducted with the probe in the air away from the test specimens. The modeled inductance was very close to the measured with difference of less than 0.3%. The difference, however, between the modeled and measured resistance was

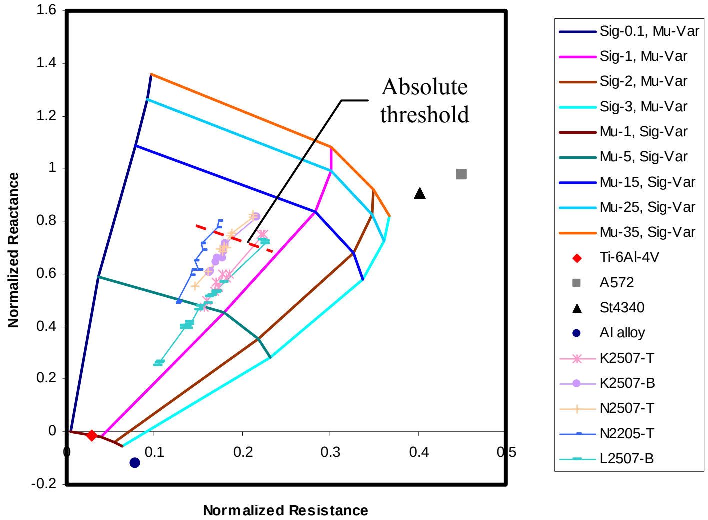


**FIGURE 3.** Simulated and measured impedance loci at 25 kHz.

systematically higher and increased with the frequency. The equivalent resistance in the model was corrected to match the measured values, since the sources of this difference (connectors, probe ferrite core, etc.) were difficult to measure, control, and account in model. Additional measures were taken to avoid the temperature and variable lift-off effects on the impedance measurements. The data was also processed during the measurement cycle to reduce the noise and random error.

Impedance-plane loci plots were modeled at four frequencies (25, 50, 100, and 200 kHz) initially (Figure 3 at 25 kHz). The $\sigma$ and $\mu_{ri}$ were varied in the range from 0.1 to 3 MS/m and from 1 to 35, respectively. The specimen measurements were clustering in three major groups as shown in Figures 3. The first group was formed from Specimens N2205-T (**T**op surface - polished). The second major group was formed from Specimens K2507-B (**B**ottom surface - polished) and N2507-T (polished). The third major group was formed from Specimens K2507-T (no machining or polishing) and L2507-B (machined).

For each of the three major groups, the relative initial magnetic permeability $\mu_{ri}$ decreased with the decrease of ferrite and increase of SP and austenite. The electrical conductivity $\sigma$ was almost constant within the group and different for each major group. The major conclusion from these initial results was that the phase change affects $\mu_{ri}$ and does not affect $\sigma$. The $\sigma$, though, was affected by the machining (except for L2507-B versus K2507-T) and chemical composition (N2205-T versus N2507-T and K2507-B).

In addition, four specimens (two nonmagnetic and two magnetic) were included for verification purposes. Based on the verification measurements and model validation in air, it was expected that the systematic (no random change of data was observed) error in estimating of $\sigma$ and $\mu_{ri}$ would fall in the range from 2 to 20%.

Once the electromagnetic properties were estimated, the eddy current spread on specimen surface and depth of penetration (DP) were also determined. The maximum surface spread was 2.3 mm while the DP was in the range from 0.3 to 0.5 mm. The standard DP formula was overestimating the modeled/actual DP approximately four times at 25 kHz and should not be used for this application. The edge effect and thickness in the model and actual specimens were not affecting the measurements.

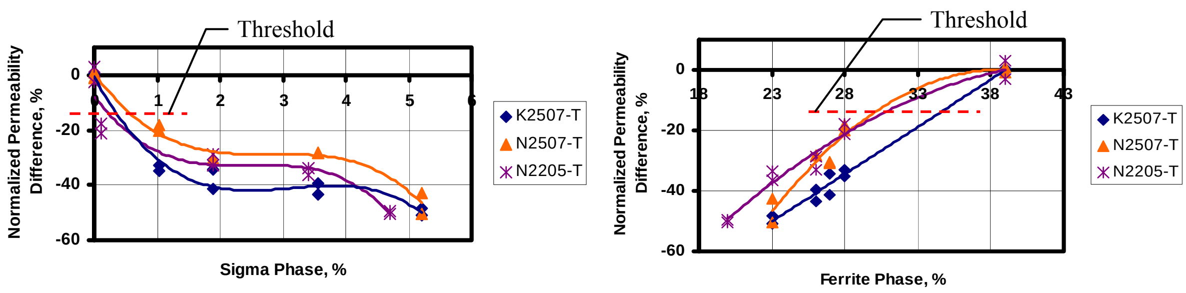


**FIGURE 4.** Normalized magnetic permeability difference versus SP and FP for K2507-T, N2507-T, and N2205-T at 25 kHz.

## Results with Advanced Eddy Current

Three representative groups with 30 measurement spots were tested at three frequencies: 25, 50, and 100 kHz. Normalized magnetic permeability difference (DNμ) was introduced (Eq. 1) to further improve the accuracy of sorting, reduce the effects of chemical composition and surface condition and also allow direct comparison of sensitivity of different methods and approaches.

$$DN\mu = \frac{\mu_j - \mu_z}{\mu_z} 100\% \tag{1}$$

where: $DN\mu$ – normalized magnetic permeability difference in percent, $\mu_z$ – average relative initial magnetic permeability measured on the specimens with minimum (**Z**ero) sigma phase content – K2507-0-T, N2507-0-T, and N2205-0-T, $\mu_j$ – relative initial magnetic permeability measured on all specimens and spots in any group (j = 1 to 10).

Typical correlation (similar for all three frequencies) between the SP and FP content and DNμ is shown in Figure 4 left and right, respectively. A decrease of DNμ was observed up to 2% increase of SP. No change in DNμ was observed between 2 and 3.5% SP. The DNμ decreased again when SP content was larger than 3.5%. A significant drop (~20%) of DNμ was observed for Specimen N2205-3-T with 0.12% sigma phase. Good correlation existed between DNμ and SP up to 2%. The change of DNμ for K2507-T reached 40 to 50% when SP changed from 0 to 2%. However, significantly more data points were required to establish the exact shape of the correlation in this important area of interest where the SP precipitation was initiated and grown up to 2%.

The FP and DNμ were strongly almost linearly correlated (Figure 4, right). The correlation was also very consistent for each group and did not change with the frequency. Since the FP was the only magnetic phase in these materials, the reduction of FP led to the reduction of magnetization properties (i.e., magnetic permeability $\mu_{ri}$) throughout the volume of the tested material clearly demonstrated in Figure 4 right.

An absolute ($\mu_{ri}$ in Figure 3) and relative (DNμ in Figure 4) threshold values can be introduced independent of specimen thickness and chemical composition.

## Results with IPA-V and IPA-S

Some limited measurements were initially taken with IPA-S because the IPA-S technique was more practical for service inspections. However, the results did not indicate good sensitivity to phase transformations and the investigation of IPA-S technique was discontinued.

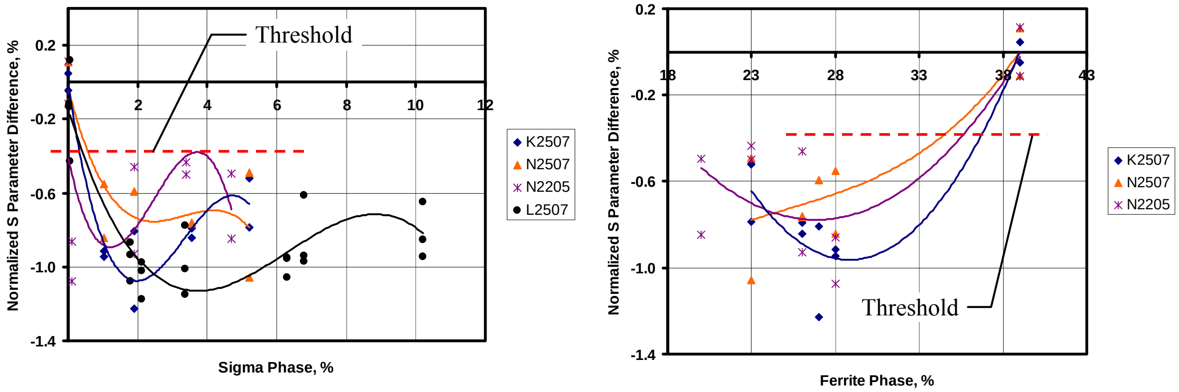


**FIGURE 5.** Normalized S parameter (IPA-V) difference versus SP and FP for K2507, N2507, N2205, and L2507.

A normalized S parameter difference (DNS) was calculated in a similar way to DNμ Eq. (1) to allow comparison of IPA-V results with the eddy current measurements of electromagnetic parameters. A relative threshold value can also be introduced independent of specimen thickness and chemical composition. The DNS parameter (Figure 5, left) decreased with the increase of SP content from 0 to 2%.

Further, the S parameter decreased with the decrease of ferrite phase (Figure 5, right). The plots shown in Figure 5 indicated that the specimens with the highest FP and lowest SP content were separated from the rest of the group. No correlation, though, existed between the DNS and FP and SP content in the entire range. The sensitivity of IPA-V method (-1% in Figure 5) was significantly lower than the sensitivity of eddy current method (-40% in Figure 4). One drawback of the IPA-V technique, if considered for service inspection, was that the specimens must be kept in a secure and protected storage for a certain period of time (2-3 days) referred to as a cooling time after the measurements until the emitted radiation drops to the natural level.

## X-Ray Diffraction Correlation to Magnetic Permeability

The correlation between the DNμ parameter and X-Ray diffraction measurements of SP content existed (Figure 6, left) and was very similar for all three frequencies. The correlation for N2507-T and N2205-T was stronger than for K2507-T. The scatter was high for all groups and particularly K2507-T. As far as the FP content was concerned, the DNμ parameter and X-Ray diffraction measurements of the FP were correlated for N2507-T and N2205-T (Figure 6, right). The data for K2507-T indicated a very weak correlation with opposite direction between the DNμ parameter and X-Ray diffraction (Figure 6, right).

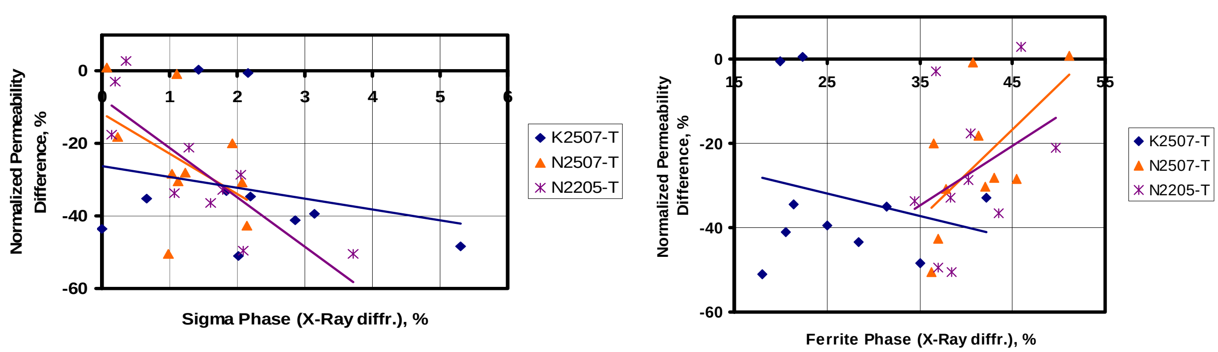


**FIGURE 6.** Normalized magnetic permeability difference versus SP (left) and FP (right) for K2507-T, N2507-T, and N2205-T at 25 kHz (both phases measured with X-Ray diffraction).

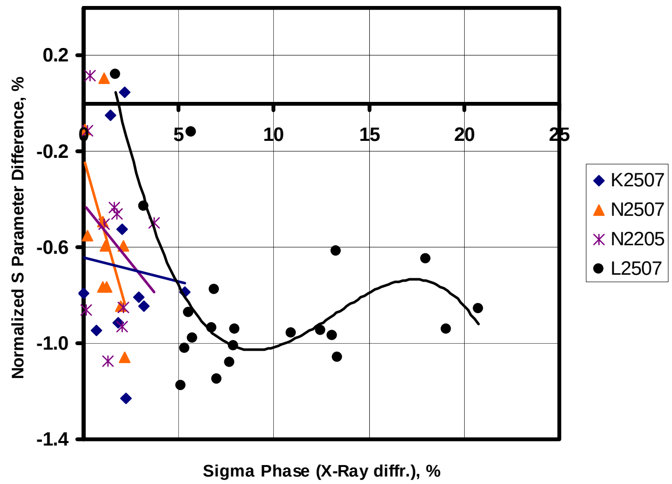


**FIGURE 7.** Normalized S parameter (IPA-V) difference versus SP (measured with X-Ray diffraction) for K2507, N2507, N2205, and L2507.

In general, the scatter of data was large. The data for K2507-T showed different pattern of behavior that might have been caused by the surface (not machined or polished) condition. The measurements also indicated that SP measurement would be possible using DNμ for the entire range obtained with the X-Ray diffraction technique. However, the establishment of more accurate correlation function is required through increased number of data points.

## X-Ray Diffraction Correlation to S-parameter

The X-Ray diffraction SP content measurements were correlated with IPA-V measurements up to approximately 5% for L2507 and 2.5% for N2205 and N2507 as shown in Figure 7. Similar to eddy current testing (Figure 6), the correlation for K2507 was weak. Again, the SP content measurement would be possible for some of the groups with IPA-V up to 5% (indicated by X-Ray diffraction test) if reliable correlation was established through increased number of data points.

## CONCLUSIONS AND RECOMMENDATION

Measurements conducted with several NDT techniques were compared to direct measurements of sigma phase (SP) acquired with X-Ray diffraction and metallographic analysis. The following conclusions can be drawn from the work on this feasibility study:

- The conventional eddy current (EC) sorting techniques were fast and reliable for screening tests and go/no-go procedures.
- The advanced eddy current technique (AECT) was used for the first time to estimate magnetic permeability ($\mu_{ri}$) and electric conductivity ($\sigma$) and link them to phase transformations for duplex stainless steels.
- The SP (some ranges) and ferrite phase (FP) content correlated well with $\mu_{ri}$ and provided high sensitivity. Threshold was possible to define regardless of material or surface condition.
- The sensitivity of induced positron analysis – volumetric (IPA-V) was smaller (approximately 40 times) than AECT.
- The X-Ray diffraction measurements were correlated with AECT and some of IPA-V measurements.
- A high scatter and surface dependence was observed for X-Ray diffraction technique.

Based on the results from this study and literature search, it was realized that identifying a reliable and practical NDT method or combination of NDT methods for SP detection and measurements would require more focused and coordinated efforts. Several recommendations can be made for future work:

- Identify and test other techniques for SP measurement if available.
- Study the relation between metallographic (volume) and X-Ray diffraction (surface) measurements.
- Focus any future studies on specimens with small SP contents.
- Increase the volume of NDT and destructive measurements to acquire statistically significant data sets.

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## REFERENCES

1. L. Karlsson, "Intermetallic Phase Precipitation in Duplex Stainless Steels and Weld Metals: Metallurgy, Influence on Properties and Welding Aspects." (Also published as: Welding Research Council Bulletin, No. 438, Jan. 1999, pp. 29, Weldasearch 192647), *Welding in the World*, **43**, No. 5, pp. 20-41 (Sept.-Oct. 1999).
2. J. Redmond and R. Davison, "Critical Review of Testing Methods Applied to Duplex Stainless Steels," *Duplex Stainless Steels '97, Proceedings, 5th World Conference*, Maastricht, Netherlands, **1**, Oct. 21-23, 1997, pp. 235-244.
3. P. Moore, *Nondestructive Testing Handbook,* third edition: **5**, *Electromagnetic Testing*, Columbus, OH, American Society for Nondestructive Testing, 2004.
4. E. I. Todorov, "Feasibility of Detection of Air Contamination in Reactive Alloy Welds with Eddy Current Methods," *ASNT's International Chemical and Petroleum Industry Inspection Technology (ICPIIT) X Conference. Paper Summaries*, Houston, TX, June 20-23, 2007, pp. 105-112.
5. E. I. Todorov, "Nondestructive Whirling (Eddy) Current Testing of Material Quality – New Methods and Means", *Masinostroene*, **XLI**, No. 9, May 1992, pp. 136-139.