COMPARISON OF TWO PROCEDURES FOR RELIABLE MEASUREMENT OF RESIDUAL STRESS IN CARBURIZED STEELS BY MAGNETIC BARKHAUSEN NOISE METHOD

Tuğçe Kaleli¹, Hüseyin Hızlı², C. Hakan Gürr ¹,³
1 Welding Technology and NDT Research/Application Center, Middle East Technical University Ankara/Turkey, e-mail: tkaleli@metu.edu.tr
2 Special Processes and Test Technologies Department, ROKETSAN Missiles Industries Inc. Ankara/Turkey, e-mail: huseyin.hizli@roketsan.com.tr
3 Metallurgical & Materials Eng. Dept., Middle East Technical University, Ankara, Turkey; e-mail: chgur@metu.edu.tr

Abstract

Sign, magnitude and distribution of residual stresses are extremely important for service performance and useful life of engineering components. Hence, monitoring their variation (qualitative approach) and measuring their magnitudes (quantitative approach) are critical issues in the manufacturing chain. Various destructive and semi-destructive methods exist to measure residual stresses; however, a rapid and reliable nondestructive method will be more appropriate for industrial applications. Recently the magnetic Barkhausen noise (MBN) method has gained importance for non-destructive determination of residual stresses in ferromagnetic materials. However, this method has some challenges due to mixed influences of residual stress and microstructure that require careful pre-calibration and verification procedures for obtaining reliable quantitative results. This paper presents the comparison of two MBN procedures for measurement of surface residual stresses in the carburized steels. Carburizing is a widely used surface treatment process that creates compressive residual stress state at the surface-near region, and thus, it remarkably improves the wear resistance and fatigue performance of low-C low-alloys steels. In the experiments 19CrNi5H and 21NiCrMo2 samples were carburized for at 900°C for different periods, and then, tempered in the range of 180°C and 600°C. In the first procedure, the MBN parameters were optimized and then MBN-r.m.s. values were correlated with the results of XRD stress measurements. In the second procedure, a pre-calibration technique based on instantaneous MBN measurements on the samples during tension/compression loading was applied. The results of two procedures were compared and discussed by focusing on reliability and applicability of the MBN method for measurement of residual stresses in industry.
1. Introduction

Engineering components are treated in order to improve their mechanical properties and service life by applying different methods. Surface treatments, such as carburizing, are one of these techniques. Similar to other thermochemical heat treatment methods, carburizing is based on variation of the surface composition. Surface hardness and wear resistance of the components are enhanced via embedding and diffusing of excess carbon into the surface of the components. The process is made up of two steps: carbon diffusion that is accompanied by the carbon gradient between core and case, and quenching which results in case hardening. As a result of these two steps, residual stresses are created at the surface-near region (1). At the end of the carburizing process, it is aimed to create compressive type residual stress on the surface that remarkably improves the wear resistance and fatigue performance of low-C low-alloys steels.

Residual stresses (RS) are defined as self-equilibrating stresses within a material in the absence of external stresses. They are local areas of tensile and compressive residual stresses and they annihilate each other to create zero force and moment within bulk material. They are caused not only by the cumulative effects of various factors in the production steps of the component but also by service conditions. The misfits between neighboring regions, created by non-uniform plastic deformation, material phase and/or density changes and surface modification are main factors in the formation of residual stresses. These stresses should be taken into account during manufacturing for service performance and useful life of engineering components. Although monitoring of their variations in a qualitative manner has been carried out by using various methods from past to present, it is difficult to determine the magnitude of the residual stresses exactly. The destructive methods provide detailed information on the distribution of the residual stress fields in the whole component; however, the component cannot be used after measurement. On the other hand, non-destructive methods maintain the efficiency and integrity of the structure, and they can be used for verification of the quality of the component. The most critical issue is to develop a rapid and reliable nondestructive method for industrial applications. The common nondestructive technique for RS measurement is X-Ray Diffraction (XRD). It adequately determines the exact RS values; however, measurement at a single point takes long time. It has also some limitations in the field measurements. Nowadays, micro-magnetic techniques can be an alternative way to determine residual stress quickly in ferromagnetic steels. The most popular and important micro-magnetic technique is magnetic Barkhausen noise (MBN) technique that is based on a movement of ferromagnetic domains in the ferromagnetic material. Transportability, short exposure times, and ability to work under severe environmental conditions are the main advantages of this technique. On the other hand, the utilization of this technique is restricted with unknown parameters since obtained data do not give an actual stress state without calibration (2).

The MBN technique is concerned with a measurement of the abrupt magnetic reorientations of the domains that are easily magnetized along a certain crystallographic direction in the ferromagnetic materials. Neighboring domains are separated from each other by domain walls. Upon application of an external magnetic field, domain walls jumps irreversibly due to discontinuous domain wall motion, nucleation and annihilation of domains. As a results of the jump of domain walls, magnetic Barkhausen noise is produced by discontinuous changes in the magnetic flux density. The Barkhausen noise
is detected as the voltage pulses induced in a pick-up coil when it is positioned near to the surface (3). These movements are influenced by several factors: microstructural features such as precipitates, grain and phase boundaries, and local stress regions. Residual stresses tend to change the area of the domain walls. Reduction in the area of 180° domain wall by compressive residual stress causes a decrease in the MBN emission (4).

It has been reported that the influence of subsequently changed elastic tensile and compressive stresses on the MBN signals was verified by XRD technique, and the peak amplitude of MBN emissions was used to correlate with both residual and applied stress. It is found a clear rising trend for the transition from compressive to tensile stress (5). In another study, the root mean square (r.m.s.) value of the MBN voltage and coercive force showed the best correlation with residual stress variations in the quenched steels (6). The residual stresses in the case hardened 18CrNiMo7 steel specimens were measured by applying both MBN and XRD method. The results showed high similarity (7). The quantitative surface residual stress values were also monitored in another research study. They tried to obtained quantitative residual stress values via MBN method with the help of constructing a special calibration curve that were obtained from tensile and compressive loading (8). Similar calibration technique was used by the other research group in order to examine variations of residual stress state in the shot peened AISI 4330 and AISI 4340 steel specimens. The residual stress values obtained from MBN measurements showed a good correlation with the results of XRD measurements (9).

The aim of this study is to investigate the applicability of the MBN method for measurement of surface residual stresses in the carburized steels and to compare two different MBN procedures for acquiring quantitative residual stress values. For this purpose, various samples were prepared and two different MBN procedures were performed; parameter optimization process and mechanical pre-calibration process. The MBN measurement results were verified with the results obtained from XRD method. Microstructural investigations and hardness measurements were also conducted.

2. Experimental Procedure

The chemical composition of the 19CrNi5H (Material A) and 21NiCrMo2 (Material B) steels are given in Table 1. The steel rods were machined into the samples with the dimensions of 165mmx40mmx10 mm. In addition, some rod-shape calibration test samples were prepared for Material B. All samples were coded with respect to the heat treatment to be applied (Table 2).

During carburizing operations of samples in group A, they were held in the furnace using the mixture of C\textsubscript{3}H\textsubscript{8} and the shielding gas (33% H\textsubscript{2}, 28% CO, 0.8% CH\textsubscript{4}) at 900°C for 13hrs. Samples in group B were held under the same atmosphere conditions, which has 1.1% C potential at 900°C for 12 hrs. Then, all samples were quenched in oil at 60°C. Finally, one sample set was left in the carburized and as-quenched condition and tempering were applied on two sample sets at 180°C and 600°C for 3 hours.
### Table 1 Chemical composition of the samples (normalized condition)

<table>
<thead>
<tr>
<th>Material</th>
<th>Chemical Composition (% weight)</th>
<th>19CrNi5H / SAE 3120</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>C  0.18</td>
<td>Si 0.26</td>
</tr>
<tr>
<td>B</td>
<td>C  0.22</td>
<td>Si 0.21</td>
</tr>
</tbody>
</table>

### Table 2 Heat-treated sample coding and heat treatment process procedures

<table>
<thead>
<tr>
<th>Sample</th>
<th>Heat Treatment Procedures</th>
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</thead>
<tbody>
<tr>
<td>AC13T0</td>
<td>13 hrs carburized at 900°C and not tempered</td>
</tr>
<tr>
<td>BC12T0</td>
<td>12 hrs carburized at 900°C and not tempered</td>
</tr>
<tr>
<td>AC13T180</td>
<td>13 hrs carburized at 900°C and tempered at 180°C for 3 hrs</td>
</tr>
<tr>
<td>BC12T600</td>
<td>12 hrs carburized at 900°C and tempered at 600°C for 3 hrs</td>
</tr>
</tbody>
</table>

MBN measurements of A-samples were performed using commercially available equipment and its software. The optimum measurement parameters were determined by considering reliability, sensitivity, validity of MBN signal with respect the residual stress levels obtained from XRD method. The optimized measurement parameters that are magnetizing voltage and magnetizing frequency were selected as 10 Volts and 250 Hz, respectively. During MBN measurements of B-samples, µScan/Rollscan 500-2 equipment was used to generate the excitation magnetic field with 125 Hz and 10 V. A standard flat-surface probe with a ferrite-cored electromagnet with a pole gap distance of 3 mm and a ferrite cored MBN pick-up fixed at the center of the pole gap was used. Calibration procedure was performed to acquire stress values from MBN emission parameter. Rod-shape calibration samples (l₀=148 mm, d₀=13 mm) representing each heat treatment condition were prepared, and were hold at 600°C for 1 hour to relieve residual stresses. Each sample had been loaded incrementally with the elastic compressive and tensile load using the universal testing machine. MBN probe was fixed on the surfaces of the samples and strain gauges was attached to the opposite side of surface of them. The surface strain values, which were obtained instantaneously, were converted to the surface stress values via Hooke’s law. Corresponding MBN parameter values were read from the device. For each heat treatment condition, surface stress values were plotted with respect to corresponding MBN parameter values to obtain calibration curves of each specimens (Figure 1).

Surface residual stresses were measured by XRD-sin²ψ method using StressTech 3000 G2/G2R equipment. Pure iron was selected as the reference material. In order to evaluate the residual stresses existed in different directions at the same region, sample was rotated by using 10 tilt the angle (ψ). Diffraction peak was specified as 156.08°. X-ray
measurements were taken from 5 different points. Microstructures were examined by an optical microscope, and micro-hardness measurements were also performed.

**Figure 1** MBN vs Stress calibration curves of the specimens for carburized, quenched and tempered samples in group B (at 900°C/12hrs)

### 3. Results and Discussion

#### 3.1. Microstructure and Hardness

Micro-hardness values of each sample were taken from both case and core region in many times. Average values of them were tabulated with standard deviation values on the Table 3. Among the same carburization condition, tempered samples have lower case hardness values than that of the carburized and as-quenched samples. Further increase in tempering temperature from 180°C to 600°C causes to the decrease in the surface hardness values due to the softening of martensite phase and spheroidization of the parent phase. The maximum hardness in the case region is 830 HV1 (58 HRC) for the AC13T0 specimen, and 820 HV1 (63 HRC) for the BC12T0 specimen. The effective case depth of carburized samples, where has approx. 550 HV1, was the same as 1.15 mm for both AC13T0 and BC12T0 specimens.

**Table 3** Results of hardness, residual stress, retained austenite and MBN measurements

<table>
<thead>
<tr>
<th>Specimen Code</th>
<th>Hardness (HV1)</th>
<th>Retained Austenite (% vol.)</th>
<th>MBN Residual Stress (MPa)</th>
<th>XRD Residual Stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AC13T0</td>
<td>835 ± 8</td>
<td>15.4±1.9%</td>
<td>-710</td>
<td>-751</td>
</tr>
<tr>
<td>AC13T180</td>
<td>730 ± 12</td>
<td>8.7±2.5%</td>
<td>-680</td>
<td>-626</td>
</tr>
<tr>
<td>AC13T600</td>
<td>364 ± 25</td>
<td>0%</td>
<td>-400</td>
<td>-339</td>
</tr>
<tr>
<td>BC12T0</td>
<td>820 ± 4</td>
<td>15%</td>
<td>-610</td>
<td>-566</td>
</tr>
<tr>
<td>BC12T180</td>
<td>750 ± 10</td>
<td>15%</td>
<td>-500</td>
<td>-515</td>
</tr>
<tr>
<td>BC12T600</td>
<td>366 ± 24</td>
<td>0%</td>
<td>-290</td>
<td>-372</td>
</tr>
</tbody>
</table>
In the metallographic examinations, micrograph of each sample was taken after the thermochemical treatment and quenching operations. They indicate that the microstructures of case regions are typically martensite and tempered martensite structures for all specimens after treatment procedures. The needle-like shape of martensite was observed in the carburized and as-quenched state. When tempering has applied at 180°C, ε-carbide precipitation occurs, martensite tends to loss its tetragonality, and it becomes to be rounded at the tip. Upon increasing the tempering temperature to 600°C, ε-carbide phase was transformed to the cementite phase and then the cementite coarsens and spheroidizes.

3.2. Residual Stresses
Residual stress values of group A samples were calculated by applying parameter optimization method. To obtain quantitative stress values, relative r.m.s. values were taken from software that were set-up with the optimum measurement parameters, and they were compared with the residual stress values obtained from the XRD measurements. When parameters were optimized correctly, MBN and XRD measurements should show good correlation with each other. Pearson’s method was applied to correlate the MBN parameter and the residual stress obtained by XRD method gave a correlation level between 0.92 and 0.94. The correlation curve for the 13hrs carburized sample given in Figure 2(10).

![Figure 2](image)

**Figure 2** Relative RMS value and residual stress correlation curve for the 13hrs carburized sample (AC13T0) (10)

For the samples in group B, residual stress values were calculated from the calibration curves (Figure 1) and tabulated on the Table 3. The results of the XRD and MBN measurements of samples are compared each other (Figure 3). It indicates that both methods gave similar results with some differences in the magnitude. It could be explained that MBN signals were collected from non-homogeneous and different phases. In addition, the information volumes of two techniques are not identical, i.e., XRD method collects the data from 10 μm-depth whereas the information depth of MBN emission is about 150 μm. In addition to differences in information depths, the probe
diameter of MBN technique is much larger than the collimator focus diameter of XRD device. The MBN probe provides the nearly 3 mm$^2$ magnetic field on the surface of specimens by using standard flat-surface probe with a pole gap distance of 3 mm whereas the smallest collimator of in 3 mm diameter was used in XRD measurement that provides information from nearly 7 mm$^2$ surface area. Hence, the both information depth and surface area are considered, the results are obtained from different information volume of specimens in XRD and MBN measurement techniques.

Figure 3 Comparison of the MBN residual stress values calculated by mechanical calibration method with the XRD surface residual stress of the carburized samples (900°C / 12hrs)

4. Conclusions

Although qualitative comparison without pre-calibration is possible, understanding of the effect of individual parameters quantitatively such as residual stress state requires some special calibration methods. In this study, firstly, optimization of magnetic parameters and then correlation between RMS values obtained from MBN measurement and the residual stress values obtained from the XRD measurements were carried out in order to predict the accurate residual stress state. Then, the mechanical calibration tension and compression tests were applied by measuring both strain and MBN emission at the surface simultaneously.

It can be said that when the MBN parameter optimization is carried out correctly, good correlation between MBN RMS values and XRD residual stress measurements should be obtained. The Pearson’s correlation factors were obtained between the range of 0.90 and 0.92. The results obtained from the mechanical calibration procedure showed the similar residual stress tendency with the XRD residual stress results. On the other hand, it was stated that there was still a considerable amount error in both two MBN measurement procedure when compared to XRD method. These variations are probably due to assumptions, different information depths of the techniques, and existence of several influential parameters in MBN measurement such as combined effects of residual stress and microstructure.
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References


