Influence of the presence of a nitrided layer on changes in the ultrasonic wave parameters

Abstract: In this work, attempts are made to estimate the relationship between the ultrasonic wave parameters and the presence of the nitrided layer. Special samples were prepared, the surfaces of which were ground and then nitrided. The samples were evaluated by ultrasonic method before and after the nitriding. During the tests, three parameters of the ultrasonic wave were recorded i.e., time of wave propagation, dominant frequency of the spectrum and bandwidth. The measurements were repeated ten times. The obtained results indicate, in particular, a reduction in the wave propagation time in the samples after nitriding compared to the samples without the nitrided layer.

Keywords: nitrided layer, ultrasonic wave

1 Introduction

In the process of manufacturing elements, nitrided layers are used on their surface. They are to improve the functional properties of the elements. There are several ways to create nitrided layers. Nitrided layers are used, for example, on the surface of crankshafts in engines or dies for extrusion of profiles [1].

After the layer has been produced, its quality is checked. One of the parameters in this evaluation is the thickness of the layer. The thickness is measured under a microscope on a metallographic specimen. This method is destructive. There are also non-destructive methods for assessing the thickness of nitrided layers. The eddy current method [2–5], Barkhausen noise [6] and ultrasonic method are used [7]. This work attempts to estimate the relationship between the ultrasonic wave parameters and the presence of the nitrided layer.

2 Nitriding of steel

Nitriding of steel is the process of saturating the surface layer with nitrogen. Nitriding is performed at an elevated temperature (usually 500–600°C) in an atmosphere containing free nitrogen atoms. Depending on the type of nitriding atmosphere, it can be distinguished into gas, ionic, powder and fluidized bed nitriding. Regardless of the type of nitriding, this process leads to the formation of a thin diffusion layer on the steel surface (Figure 1), which significantly increases the durability of the detail, and thus also of the entire technical objects.

The nitrided layer is characterized by high hardness and resistance to abrasion, corrosion and fatigue. The structure, thickness and properties of the nitrided layer depend on the type of steel and the parameters of the nitriding process itself – temperature, nitrogen potential and time [8].

Gas nitriding is the most commonly used in industrial technology. Depending on the nitriding atmosphere used, gas nitriding is distinguished into one-component (ammonia) or two-component nitriding (ammonia and dissociated ammonia or ammonia and nitrogen) [9].

3 Methods of assessing the nitrided layer

3.1 Eddy current and magnetic Barkhausen noise method

In the works [2–5], attempts were made to use the eddy current method to assess the thickness of the nitrided layers. Samples made of 38HMJ and WCL steel were tested. The dependence of the measuring device signal
on the thickness of nitrided layers on the previously prepared samples was investigated. Wirotest measuring equipment was used for the tests, together with sensors with different magnetization current frequencies. The conducted research has shown that this method can be successfully used to assess the thickness of the nitrided layer. A sensor was used to measure the thickness of the nitrided layer with a magnetization current frequency of 3.4 kHz. For both 38 HMJ and WCL steels, a high correlation was obtained between the indications of the measuring instrument and the thickness of the nitrided layer ($R^2$ coefficient values at the level of 0.93–0.99). The parameters of the performed nitriding processes influenced the level of correlation, but in all cases, the indications of the measuring instrument increased along with the increase in the thickness of the nitrided layer (Figure 2).

In the literature, it is possible to write works in which the Barkhausen noise method were used for the assessment of nitrided layers. Stupakov et al. [6] state that the classical RMS parameter demonstrated the best sensitivity to growing thickness of the hardened surface layer. The commercial device with the small attachable sensor was able to estimate the nitrided case of up to 100–150 µm thickness.

### 3.2 Ultrasonic method

Ultrasonic method waves have great potential in the assessment of nitrided layers. In particular, Rayleigh waves and Love waves. Unfortunately, there are very few studies on this subject. Belahcene et al. [7] used surface waves of different propagation depths. The authors investigated an aircraft engine bearing a nitrided surface. A measuring system with two heads was used for the tests (Figure 3). The distance between the transmitter and receiver was constant. Waves with frequencies from 3 to 12 MHz were introduced, which resulted in a different depth of wave penetration from 0.25 to 0.85 mm.

According to the authors, the thickness of the layer is defined by the wavelength above which the plateau is observed in the variability of wave propagation. The works undertaken by the authors were not continued.

Surface waves were also used by Samolczyk and Baer [10]. In this case, the layer thickness was determined on the basis of the determined calibration curve. To determine the calibration curve, samples of WCL steel subjected to ion nitriding at a temperature of 500°C were prepared. USLT 2000 flaw detector and 4 MHz MBW 90-4E heads were used for the measurements. The calibration curve was based on changes in the velocity of the surface wave. In this case also, the authors did not continue their work.

The use of the Love wave should provide great possibilities in the assessment of nitrided layers. This wave

---

**Figure 1:** Nitrided layer on 40 HM steel [7].

**Figure 2:** The relationship between the thickness of the nitrided layer on 38 HMJ steel and the mean readings of the Wirotest 03 instrument [5].

**Figure 3:** The measuring system on the bearing surface [7].
propagates in the layers created on the surfaces of objects. This wave is highly dispersive, which means that its speed depends on the change in material properties [11–13]. One of the methods of its production consists in the use of a blade head [14], laser [15] or Hertzian’s head [16]. In the experiment described in this article, a blade head was used.

4 Experimental methods

4.1 Purpose and scope of the experiment

The aim of the experiment was to check the sensitivity of changes in the parameters of the ultrasonic wave to the presence of the nitrided coating. The scope of the experiment included the preparation of samples on which the ultrasonic wave parameters were measured. Then, the samples were subjected to the nitriding process. After forming the nitriding layer, measurements of the ultrasonic wave parameters were performed. The parameters of the wave on the samples before and after nitriding were compared.

4.2 Research object

Samples from steel 38 HMJ were used for the tests (Figure 4). It is a structural chromium–molybdenum steel intended for nitriding. The chemical composition of steel is given in Table 1.

The gas nitriding process in an ammonia atmosphere lasted 19 h at a temperature of 550°C with a nitrogen potential $N_p$ of 3.0. The view of the samples after nitriding is shown in Figure 5.

4.3 Measuring system and methodology of research

An ultrasonic flaw detector UMT-12 and a UZIP blade head with a frequency of 4 MHz were used to perform the measurements (Figure 6). The head consists of two transducers. One is the transmitter and the other is the receiver of the wave. The wave is introduced into the object and picked up by blades connected to the transducers.

In the first stage of the research, the probe was applied to the non-nitrided samples. The ultrasonic wave that reached the receiver is shown in Figure 7. The pulse of the received wave was subjected to the Fourier transform and the amplitude–frequency spectrum was obtained (Figure 8).

Three ultrasonic parameters were analyzed during the research – time of wave propagation $t$, dominant frequency of the spectrum $f_{\text{max}}$ and bandwidth $B$ (Figure 9).

![Figure 4: The samples used during the tests are marked with letters A–F.](image1)

![Figure 5: View of samples after the nitriding process.](image2)

| Table 1: Chemical composition of steel 38 HMJ [17] |
|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| C      | Si     | Mn     | Cr     | Mo     | Al     | Ni     | V      | W      | S      | P      |
| 0.35–0.42 | 0.17–0.37 | 0.3–0.6 | 1.35–1.65 | 0.15–0.25 | 0.7–1.1 | max 0.25 | max 0.05 | max 0.2 | max 0.025 | max 0.025 |
Each sample has two flat surfaces (top – 1 and bottom – 2). An ultrasonic head was attached to each of them and the measurements were repeated ten times. The measurements were made with the head blades with spacing of 50 mm and with the constant pressure of the head to the surface, which was provided by a constant mass weight. A total of 120 measurements were performed on the samples before nitriding and 120 measurements on the samples after nitriding. In each application, three parameters of the ultrasonic wave were measured.

4.4 Results of research

Detailed results of measurements for sample A before and after nitriding are presented in Tables 2 and 3. Based on the obtained results, their mean value $\bar{x}$ and standard deviation $s$ were calculated. Meanwhile, the mean values and standard deviations for all samples are presented in Tables 4 and 5.

The summary of the mean values for the samples A–F (sides 1 and 2) before and after nitriding is shown in Figures 10–12, respectively, for the ultrasonic wave propagation time $t$, dominant frequency of the spectrum $f_{max}$ and bandwidth $B$.

4.5 Analysis of research

Comparing the results of the measurement of the mean values for the ultrasonic wave propagation time $t$ for the samples before nitriding, it can be seen that these values are identical. The mean values are 36.50 $\mu$s and are the same for all samples A through F. The standard deviation
Parameter of the amplitude–frequency spectrum: $f_{\text{max}}$ – dominant frequency of the spectrum (MHz) and $B$ – bandwidth (MHz).

**Table 2:** Results for sample A before nitriding

<table>
<thead>
<tr>
<th>No.</th>
<th>t (µs)</th>
<th>$f_{\text{max}}$ (MHz)</th>
<th>$B$ (MHz)</th>
<th>t (µs)</th>
<th>$f_{\text{max}}$ (MHz)</th>
<th>$B$ (MHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>39.50</td>
<td>3.80</td>
<td>1.73</td>
<td>39.50</td>
<td>3.80</td>
<td>1.83</td>
</tr>
<tr>
<td>2</td>
<td>39.50</td>
<td>3.73</td>
<td>1.76</td>
<td>39.50</td>
<td>3.80</td>
<td>1.73</td>
</tr>
<tr>
<td>3</td>
<td>39.50</td>
<td>3.80</td>
<td>1.83</td>
<td>39.50</td>
<td>3.80</td>
<td>1.76</td>
</tr>
<tr>
<td>4</td>
<td>39.50</td>
<td>3.80</td>
<td>1.83</td>
<td>39.50</td>
<td>3.80</td>
<td>1.83</td>
</tr>
<tr>
<td>5</td>
<td>39.50</td>
<td>3.73</td>
<td>1.76</td>
<td>39.50</td>
<td>3.87</td>
<td>1.69</td>
</tr>
<tr>
<td>6</td>
<td>39.50</td>
<td>3.76</td>
<td>1.76</td>
<td>39.50</td>
<td>3.76</td>
<td>1.76</td>
</tr>
<tr>
<td>7</td>
<td>39.50</td>
<td>3.73</td>
<td>1.83</td>
<td>39.50</td>
<td>3.73</td>
<td>1.83</td>
</tr>
<tr>
<td>8</td>
<td>39.50</td>
<td>3.80</td>
<td>1.83</td>
<td>39.50</td>
<td>3.80</td>
<td>1.69</td>
</tr>
<tr>
<td>9</td>
<td>39.50</td>
<td>3.73</td>
<td>1.83</td>
<td>39.50</td>
<td>3.73</td>
<td>1.83</td>
</tr>
<tr>
<td>10</td>
<td>39.50</td>
<td>3.80</td>
<td>1.76</td>
<td>39.50</td>
<td>3.80</td>
<td>1.83</td>
</tr>
<tr>
<td>$\bar{x}$</td>
<td>39.50</td>
<td>3.77</td>
<td>1.79</td>
<td>$\bar{x}$</td>
<td>39.50</td>
<td>3.79</td>
</tr>
<tr>
<td>s</td>
<td>0</td>
<td>0.035</td>
<td>0.041</td>
<td>0</td>
<td>0.041</td>
<td>0.060</td>
</tr>
</tbody>
</table>

$\bar{x}$ - mean value, s - standard deviation.

**Table 3:** Results for sample A after nitriding

<table>
<thead>
<tr>
<th>No.</th>
<th>t (µs)</th>
<th>$f_{\text{max}}$ (MHz)</th>
<th>$B$ (MHz)</th>
<th>t (µs)</th>
<th>$f_{\text{max}}$ (MHz)</th>
<th>$B$ (MHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>22.94</td>
<td>3.73</td>
<td>1.76</td>
<td>22.94</td>
<td>3.66</td>
<td>1.76</td>
</tr>
<tr>
<td>2</td>
<td>22.94</td>
<td>3.66</td>
<td>1.83</td>
<td>22.94</td>
<td>3.73</td>
<td>1.76</td>
</tr>
<tr>
<td>3</td>
<td>22.94</td>
<td>3.73</td>
<td>1.83</td>
<td>22.94</td>
<td>3.73</td>
<td>1.73</td>
</tr>
<tr>
<td>4</td>
<td>22.94</td>
<td>3.73</td>
<td>1.83</td>
<td>22.94</td>
<td>3.66</td>
<td>1.90</td>
</tr>
<tr>
<td>5</td>
<td>22.94</td>
<td>3.73</td>
<td>1.90</td>
<td>22.94</td>
<td>3.73</td>
<td>1.83</td>
</tr>
<tr>
<td>6</td>
<td>22.94</td>
<td>3.73</td>
<td>1.83</td>
<td>22.94</td>
<td>3.66</td>
<td>1.76</td>
</tr>
<tr>
<td>7</td>
<td>22.94</td>
<td>3.80</td>
<td>1.76</td>
<td>22.94</td>
<td>3.73</td>
<td>1.76</td>
</tr>
<tr>
<td>8</td>
<td>22.94</td>
<td>3.73</td>
<td>1.76</td>
<td>22.94</td>
<td>3.73</td>
<td>1.69</td>
</tr>
<tr>
<td>9</td>
<td>22.94</td>
<td>3.80</td>
<td>1.76</td>
<td>22.94</td>
<td>3.73</td>
<td>1.69</td>
</tr>
<tr>
<td>10</td>
<td>22.94</td>
<td>3.80</td>
<td>1.76</td>
<td>22.94</td>
<td>3.73</td>
<td>1.76</td>
</tr>
<tr>
<td>$\bar{x}$</td>
<td>22.94</td>
<td>3.74</td>
<td>1.80</td>
<td>$\bar{x}$</td>
<td>22.94</td>
<td>3.71</td>
</tr>
<tr>
<td>s</td>
<td>0</td>
<td>0.044</td>
<td>0.049</td>
<td>0</td>
<td>0.034</td>
<td>0.062</td>
</tr>
</tbody>
</table>

$\bar{x}$ - mean value, s - standard deviation.

The mean dominant frequency of the spectrum $f_{\text{max}}$ is 0.0. For the same samples (before nitriding), the mean dominant frequency of the spectrum $f_{\text{max}}$ values are also very similar, ranging from 3.73 to 3.79 MHz with a standard deviation of 0.023–0.062 MHz. The standard deviation of 0.062 MHz is only 1.6% of the value of 3.73 MHz. It is also similar to the average values of the bandwidth $B$, whose values ranged from 1.70 to 1.79 MHz, with a standard deviation of 0.041–0.097. The standard deviation of 0.097 MHz is 5.7% of the value of 1.70 MHz. It can be concluded that in the samples before nitriding, the dispersion of the values of all three parameters of the ultrasonic wave is small. The values are stable and repeatable.
After the nitriding process was performed, the values of the measured ultrasonic wave parameters changed. The mean values of ultrasonic wave propagation time $t$ are 22.94 $\mu$s and are the same for all samples A through F. The standard deviation is 0.0. The mean dominant frequency of the spectrum $f_{\text{max}}$ is from 3.71 to 3.75 MHz with a standard deviation of 0.034–0.056 MHz. The standard deviation of 0.056 MHz is only 1.5% of the value of 3.71 MHz. The average values of the bandwidth $B$ is from 1.75 to 1.80 MHz, with a standard deviation of 0.034–0.090. The standard deviation of 0.090 MHz is 5.1% of the value of 1.75 MHz. It can be concluded that the dispersion of the values of all three parameters of the ultrasonic wave in the samples after nitriding is small. The values are stable and repeatable, similar to the samples before nitriding.

The stability and reproducibility of the results of all three parameters in the samples before and after nitriding means that the samples were homogeneous in terms of ultrasonic wave propagation in them. The homogeneity was before and after the nitriding process.

While comparing the differences in the values of individual parameters in the samples before and after nitriding,
it should be noted that the average wave propagation time $t$ decreased from 39.50 to 22.94, by as much as 16.56 $\mu$s. The reduction in the wave transit time, for the same length of its path, means that there was an increase in the wave propagation speed in the samples after nitriding. The increase in velocity, therefore, had to be due to the presence of a nitrided layer. Thus, it should be stated that the wave velocity is sensitive to the presence of the nitrided layer.

In the case of the mean dominant frequency of the spectrum $f_{\text{max}}$ value, the changes are not as significant as in the case of the ultrasonic wave propagation time $t$. However, it can be observed that for each of the 12 surfaces (from A1 to F2), the dominant frequency of the spectrum $f_{\text{max}}$ values on the samples after nitriding are lower than the values on the samples before nitriding. This can be seen very clearly in Figure 11.

A similar relationship can also be indicated in the values of the bandwidth $B$. Changes in the value of this parameter on the samples after nitriding are not so significant; however, on the surface after nitriding (except for A2 and B1 surfaces), the value of this parameter is higher than in the case of surfaces before nitriding. This relationship is clearly visible in Figure 12.

5 Conclusion

The obtained results show the high repeatability of the ultrasonic wave parameters propagating values in the samples without the nitrided layer and in the samples with the nitrided layer. The value of one of the parameters changed significantly after the formation of the nitrided layer. This parameter is the wave propagation time which results from the change in the velocity of the wave. All samples were subjected to the same nitriding process, so the layer produced in them was identical in terms of thickness. Thus, the presence of this layer caused an identical change in the wave propagation time in all samples, i.e., on all 12 surfaces.

The conducted research was reconnaissance and initial. The observed changes in the wave propagation time due to the presence of the nitrided layer allowed for further research to be planned. They should be aimed at measuring the wave propagation time in samples with different nitrided layer thicknesses.

Funding information: Research work was financed by the project 0414/SBAD/3612.

Conflict of interest: Author state no conflict interest.

References


