MATERIALS CHARACTERISATION

Steel hardness evaluation based on ultrasound velocity measurements

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Material properties such as strength, toughness or hardness are usually determined by destructive tests. However, continuous destructive measurements are generally difficult to perform during the production process, which creates a need for a fast and easy non-destructive method of material characterisation.

Material elastic parameters, such as Young's modulus, bulk modulus etc, can be directly evaluated using non-destructive methods based on the measurements of ultrasonic wave velocity (shear and longitudinal). However, strength or hardness cannot be determined directly from those parameters since they also depend on material microstructure, such as grain size and orientation, as well as other factors.

This paper presents an experimental evidence that hardness of rolled martensitic steel can be evaluated based on the measurements of ultrasonic wave velocity and limited information of its chemical composition.

1. Introduction

Common applications of ultrasound to NDT are concerned with the detection and characterisation of material flaws or measurement of material thickness. Ultrasonic measurements can also be used for the characterisation of material properties; parameters, such as elastic modulus, material microstructure, hardness etc, can be estimated from the ultrasonic measurements[1,2]. Elastic modules (Young's modulus, bulk modulus, Poisson's ratio etc) can be directly calculated from the shear and longitudinal wave velocities. Strength, toughness or hardness, which are determined by the chemical composition and thermal treatment of steel products, can be indirectly inferred from the ultrasonic measurements using theoretical relationships and empirical correlations.

Standard destructive hardness tests require taking samples, which is time consuming and expensive during an in-line production. If a high accuracy is needed, the instruments used for this kind of test are generally bulky and heavy. Light hand-held testers are also available but they have generally much lower accuracy or they need to be calibrated for each material separately. Even portable non-destructive testers have been developed, for instance the instruments based on the ultrasonic contact impedance method[3]. When correctly calibrated for the specific material under test, an ultrasonic contact impedance instrument is capable of providing correct results expressed in all hardness scales (Rockwell, Vickers, Brinell or Shore). However, its operation requires a physical contact with the surface of the tested sample. Also, testers that combine a few methods, such as the ultrasonic contact impedance and rebound method, have been developed in order to increase reliability of the results[4].

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Another non-destructive solution is to estimate hardness level based on the correlation between ultrasound wave velocity and attenuation with the real hardness values measured using traditional methods. Papadakis[1] described an experiment carried out by Yee in 1971 where an ultrasonic method for evaluating an average hardness in structures made of D6ac steel was used. Their method was based on the velocity measurement of surface waves and an assumption that the surface wave velocity depends on hardness in the same way as the longitudinal velocity, which had been established by the authors previously. Another result, presented in[2], proves that material hardness can be determined only by the measurements of ultrasound wave velocity and attenuation. The measurements showed a parabolic relationship between hardness and ultrasonic velocity, while attenuation changed linearly along with hardness. The relationship between steel hardness and ultrasound velocity and attenuation were also presented in the literature[5].

In each of the reports mentioned above, the results were obtained for one specific type of material only, and various materials showed different relationships between hardness and ultrasound velocity. In a collaborative project, colleagues at Swerea KIMAB have shown that the ultrasound velocity of longitudinal waves can be used to determine the hardness of different types of martensitic steel without the need for adjusting the fitting curve in each case[6]. We have developed this idea further by using shear waves and also including chemical analysis in the model to predict hardness.

In this paper, an experimental evidence is presented that ultrasound wave velocity measurements can be used to determine the hardness of different types of martensitic steel, without the need for adjusting the fitting curve in each case.

To establish the correlation with hardness, precise ultrasonic velocity must be measured. Although attenuation measurements may be more sensitive to changes in material microstructure, due to the simplicity of measurement, sound velocity and chemical analysis were used in this study to predict hardness.

2. Ultrasonic measurements

The most commonly used technique for measuring velocity of ultrasonic bulk waves is the pulse-echo method. An ultrasonic transducer is used as a transmitter and receiver and a number of multiple echoes, reflected from the bottom of the sample, are recorded. If the sample thickness is known, ultrasonic wave velocity can be directly calculated from the time-of-flight (TOF) estimated using successive pulse peaks or zero-crossings[7]. These methods, however, may suffer from poor resolution, especially when an attenuated ultrasonic pulse is sampled with a low sampling rate. To improve the resolution, simple signal processing methods can be applied, for example zero-crossing interpolation or pulse correlation.

If backwall echoes are digitised, the linear interpolation of zero-crossing points[8] can be used to accurately evaluate TOF. The zero-crossing point is determined as an intersection of the horizontal zero line and the interpolated linear function. It is worth noting that, although this method results in a high time resolution, its

inaccuracy still depends on the precision of the sampling operation of the analogue-to-digital converter used for digitising analogue signals.

For dispersive media, where phase and group velocities are frequency dependent, the spectral phase method can be used. The velocities can be determined from the phase spectra of two subsequent echoes. The spectra can be calculated using the discrete Fourier transform. More information concerning velocity estimation methods can be found in the literature[8,9].

3. Experiment

Measurements of the longitudinal wave velocity were performed in immersion, in the measurement set-up shown in Figure 1, using a 3.5 MHz piezoelectric immersion transducer from Panametrics connected to the ultrasonic board USPC3100LA from Socomate International, France. The USPC board was provided with the LabView user interface and its sampling frequency was set to 100 MHz. To increase the accuracy of the TOF measurement, six successive bottom echoes were recorded in each measurement. The data were digitised and stored for further processing. Zero-crossing interpolation[8] was implemented to estimate the TOF from the backwall echoes. This step improved resolution of the time measurements to 0.1 ns.
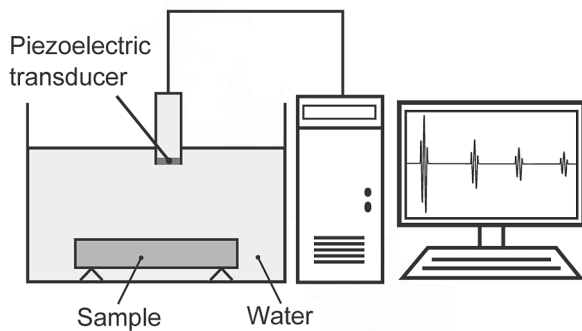

Figure 1. Longitudinal wave measurement set-up

Measurement accuracy in the estimation of longitudinal and shear velocities was calculated as a geometrical sum of the uncertainties due to the time and thickness measurement resolution:

$$\Delta c = \sqrt{\left(\frac{\partial c}{\partial l}\Delta l\right)^2 + \left(\frac{\partial c}{\partial t}\Delta t\right)^2} = \sqrt{\left(\frac{1}{t}\Delta l\right)^2 + \left(\frac{l}{t^2}\Delta t\right)^2} \quad ....(1)$$

where Δl and Δt denote thickness and time measurement accuracy, respectively.

The longitudinal velocity accuracy estimated using Equation (1) was between 3.7 and 9.7 m/s (depending on the specimen thickness).

The shear wave velocity measurements were carried out in the set-up shown in Figure 2. A polarised EMAT probe from Sonemat Ltd, UK, was used to measure shear wave velocity in pulse-echo mode. Steel plate specimens were placed under the EMAT transducer (since an EMAT contains a strong permanent magnet it attracts ferromagnetic steel). The EMAT was connected to the high-voltage pulse generator in the Ritec RAM-5000 through the Ritec diplexer. The signal received from the diplexer was amplified approximately 60 dB by one of the RAM-5000 receivers. The Agilent Inniium digital oscilloscope with sampling frequency 500 MHz was used for the TOF measurements.

Similarly to the longitudinal wave measurements, multiple echoes were recorded to increase accuracy. Ten echoes were time averaged and stored at the Agilent oscilloscope for each time measurement, which was performed directly at the instrument's screen using the zero-crossing method. The oscilloscope automatically performed filtration and interpolation of the signal, which resulted in the
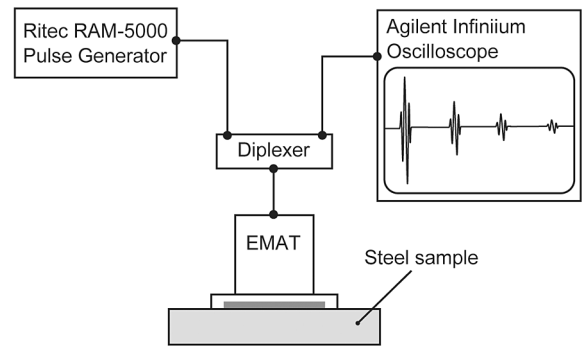

Figure 2. Shear wave measurement set-up

improved resolution in the time measurements of 0.01 ns. Due to the slower velocity and higher sampling frequency, the accuracy obtained during shear wave measurements was better than that for the longitudinal velocity and was between 1.6 and 4.3 m/s.

Ultrasonic measurements were performed on 17 specimens, which represented five types of high-quality martensitic steel with hardness and thickness as specified in Table 1. The specimen thickness was measured mechanically using a micrometer with an accuracy of 0.01 mm.

The experiment consisted of several steps where hardness of the specimens was gradually modified by thermal treatment (tempering). The successive thermal treatment steps consisted of 30 minutes' tempering at the temperatures specified in Table 1. It can be seen from Table 1 that the specimens had different initial hardness values and their hardness decreased after each tempering step. The specimen's hardness was evaluated using a Brinell Hardness Tester after each tempering step, before the ultrasonic wave velocity measurements were performed. Each time, hardness was measured in three different places and then averaged. It is worth noting that the test was 'blind', which means that neither the steel chemical analysis nor the specimen hardness were revealed to the researcher measuring the velocity during the test.

Multiple linear regression[10] analysis was performed after completion of all tempering steps. Ultrasonic wave velocities were correlated with the samples' hardness and other parameters, such as content of chemical elements and tempering temperature. The correlation coefficient R^2 and the root mean square error (RMSE) were used as a measure of the relationship between those parameters.

4. Results

Results of the velocity measurements for all the inspected specimens are presented in Figures 3(a) and 3(b). The bars corresponding to each specimen show the change of shear (Figure 3(a)) and longitudinal (Figure 3(b)) wave velocities after each tempering step. It is apparent that the velocities corresponding to all specimens tend to increase after each tempering step. Note that the velocity measured for the specimen G (the only sample of steel AR600) is considerably lower than the velocities corresponding to other specimens. A comparison of Figure 3 and Table 1 implies an inverse proportional relationship between the ultrasonic velocity and hardness.

Results presented in Figures 4(a) and 4(b) show a correlation between hardness and ultrasonic wave velocities for shear and longitudinal velocity, respectively. The velocity shows a slightly non-linear relationship to hardness, which confirms the evidence mentioned above[2]. In Figure 4, the relationship has been approximated using parabolic curves corresponding to different steel types. It is clearly visible that the shear velocity shows better correlation with hardness than the longitudinal velocity.

It is apparent that the correlation between velocity and hardness depends on the steel type. Even though shear velocity for four

Table 1. Steel types, hardness and thickness

| Sample mark	Steel type	Hardness [HB] corresponding to tempering temperature						Thickness [mm]
		Not tempered	200°C	300°C	400°C	500°C	600°C	
A	AR550	547	542	480	445	378	305	18.35
B	AR550	546	559	479	440	371	307	9.54
C	AR550	536	540	485	450	382	311	19.66
D	AR500	497	507	458	424	347	284	15.11
E	AR450	451	453	428	376	294	255	7.60
F	AR500	510	516	472	421	332	278	11.37
G	AR600	586	585	512	437	362	303	14.16
H	AR500	513	504	458	402	313	263	7.60
I	AR450	465	470	449	401	313	273	11.37
J	AR400	415	417	405	361	278	240	7.62
K	AR400	396	403	405	379	298	255	14.13
L	AR400	409	414	415	384	310	260	19.09
M	AR400	407	409	412	375	296	259	14.04
N	AR500	507	509	472	430	358	292	14.05
O	AR450	459	465	449	403	315	272	11.34
P	AR450	455	460	454	418	326	277	15.24
R	AR450	451	453	451	416	332	274	19.08

types of steel is similar and the correlation for them could be pleasingly represented by only one curve, this does not apply to specimen G, representing the fifth steel type. The correlation of longitudinal velocity shows even worse results in terms of higher scattering around the fitting curves.

It is apparent that specimens' chemical composition and tempering temperature, that vitally influence their hardness, should be taken into consideration in the correlation analysis. To calculate this relationship, a multiple linear regression was performed including 16 parameters: 14 chemical elements, the tempering temperature and the velocity. The regression results for shear and longitudinal velocity are presented in Figures 5(a) and 5(b), respectively. Numerical values of the correlation coefficients R^2 and the RMSE for hardness, calculated for shear and longitudinal wave velocity, are presented in Table 2.

In the experiment reported here, both chemical composition and thermal treatment of our specimens were strictly controlled. During an in-line process, however, it is generally impossible to determine the exact temperature and length of the thermal treatment in the material flow. To get more insight into how the thermal treatment affects obtained results, multiple linear regression
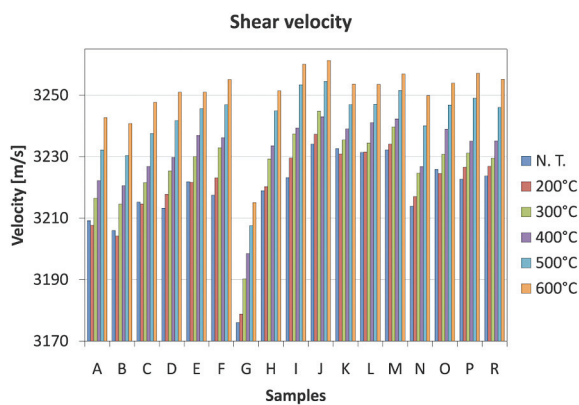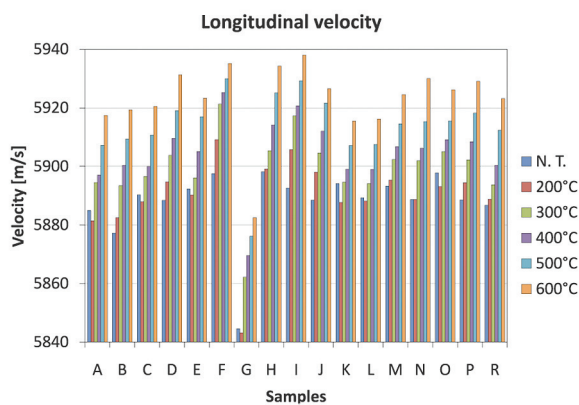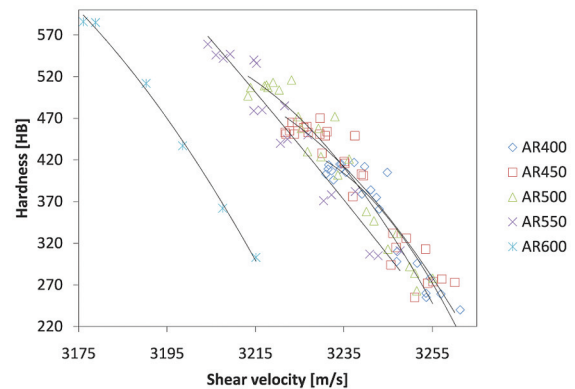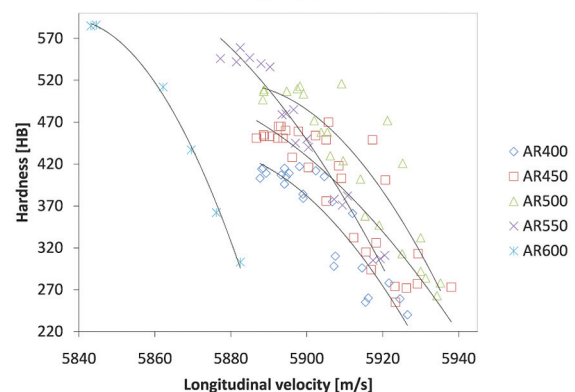

Figure 3. Velocity change during tempering process: (a) shear velocity, (b) longitudinal velocity. The bars corresponding to the successive tempering steps differ in colour
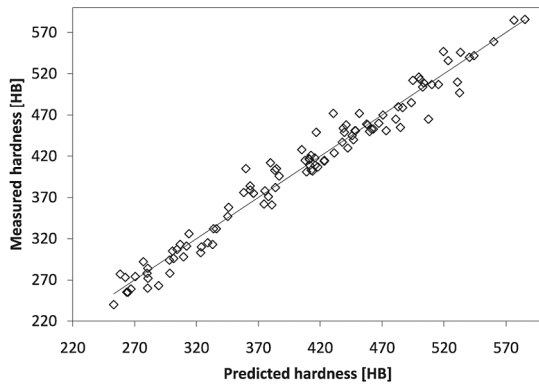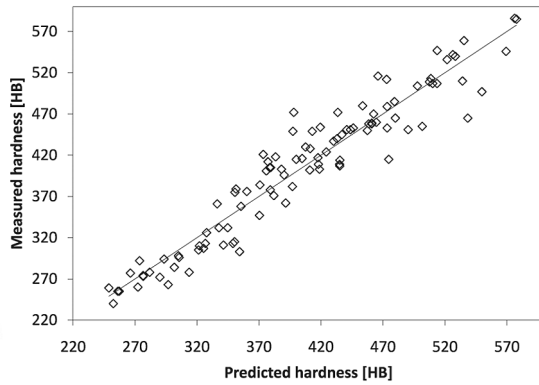

Figure 4. Correlation between hardness and velocity calculated for shear velocity (a) and longitudinal velocity (b)

(a)

(b)

Figure 5. Hardness prediction using multiple regression taking into account velocity, tempering temperature and 14 chemical elements corresponding to shear velocity (a) and longitudinal velocity (b)

analysis was carried out for a reduced number of parameters. The independent variables used in the regression analysis included complete chemical analysis data available from the steel manufacturer and two cases were considered separately, with the tempering temperature included or not.

It appeared that the temperature, along with complete chemical composition of the tested steel, had a very small influence on the results. Although the calculated correlation coefficients turned out to be worse, the differences were approximately 1-3% of the R^2 value, and the RMSE difference was up to 0.5 [HB] for both shear and longitudinal velocity (see Table 2).

Table 2. Results of the multiple regression

	v_s		v_l	
	R^2	RMSE [HB]	R^2	RMSE [HB]
14 chem el + temp	0.969	16.5	0.917	27.2
14 chem el	0.966	17.2	0.916	27.2
Nickel + temp	0.929	23.5	0.747	44.3
Nickel	0.928	23.5	0.641	52.5
Chrome + temp	0.890	29.15	0.849	34.1
Chrome	0.761	42.8	0.682	49.4
5 chem el	0.934	23.0	0.858	33.6

However, to get more insight into the problem and possibly to reduce the amount of variables, we searched the most significant chemical element that could be used for predicting hardness changes without a considerable decrease in accuracy. To do that, linear regression was applied for every single chemical element and velocity, either with or without temperature.
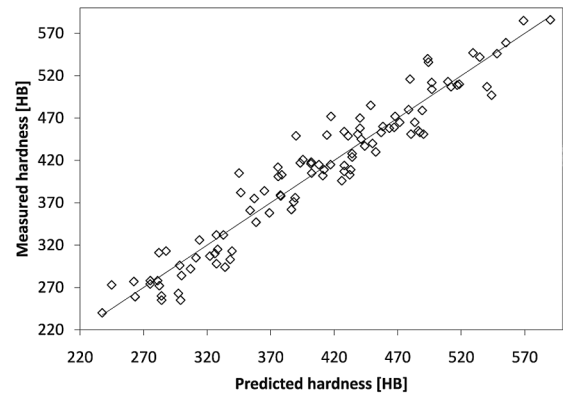
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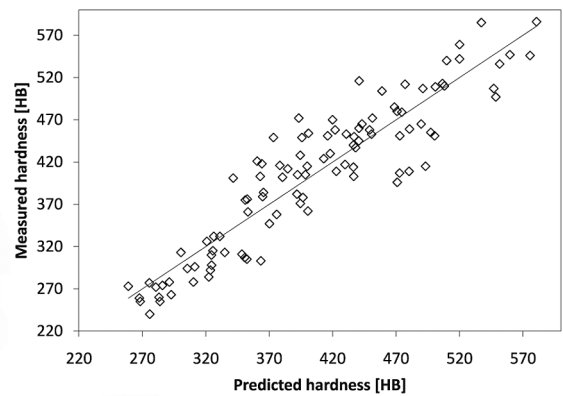
(b)

Figure 6. Hardness prediction for shear velocity, temperature and nickel (a), and longitudinal velocity, temperature and chrome (b)

Nickel (Ni) appeared to be the most signicant for predicting hardness using shear wave velocity. Ni content in the steel, together with the tempering temperature and velocity, resulted in accurate estimates of hardness level (see Figure 6(a) and Table 2), and the correlation value R^2 without tempering temperature was only 1% lower than when it was included.

It appeared, however, that for longitudinal velocity, nickel content together even with temperature and velocity did not produce accurate results of hardness level. Another chemical element that, combined with those parameters, yielded much better results was chrome (Cr). Along with the temperature and velocity, it correlates with hardness as shown in Figure 6(b) and Table 2. Unfortunately, without tempering temperature, no chemical element alone produces accurate results along with the longitudinal velocity.

A possible solution to the problem of temperature uncertainty is to use five chemical elements in the regression model. It appeared that selection of the significant elements was a hard task since the number of possible data combinations is very large. After some preliminary calculations and physical reasoning, the following elements have been chosen: nickel (Ni), chrome (Cr), silicon (Si), molybdenum (Mo) and manganese (Mn). Results obtained for this model are presented in Figure 7 and the lowest row of Table 2.

5. Conclusions

The relationship between hardness and ultrasonic wave velocity was investigated for 17 specimens of high-quality rolled martensitic steel. It was shown that material hardness and ultrasonic velocity are correlated: the velocity decreases with hardness. Predicting hardness based on wave velocity is possible but it requires separate relations for different steel types.

It was also shown that when additional information about
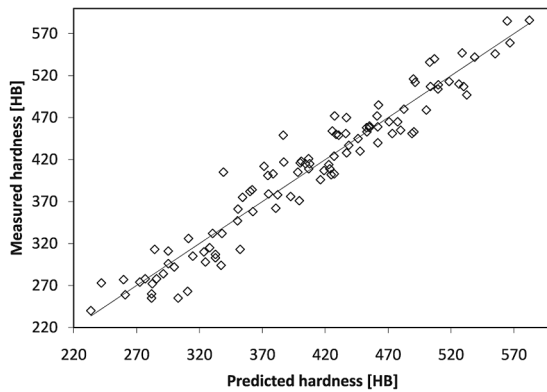
(a)
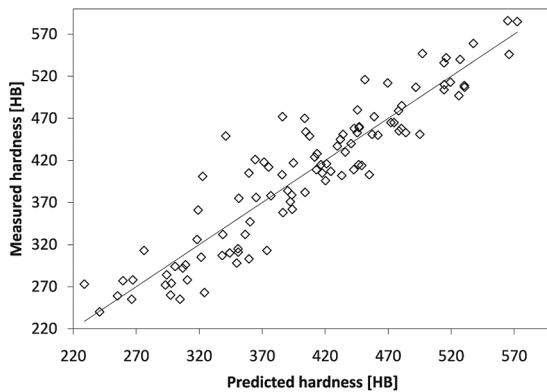
(b)

Figure 7. Hardness prediction for five chemical elements and shear velocity (a), and longitudinal velocity (b)

the material, such as its chemical composition and tempering temperature, is used in the multiple regression, high correlation between ultrasonic wave velocity and hardness can be obtained for different types of steel. Essentially, steel hardness can be predicted based on the shear wave velocity provided that content of only one chemical element is known. For longitudinal wave velocity, knowledge of tempering temperature is essential when considering only one chemical component. However, when the conditions of thermal treatment are unknown, at least five chemical elements should be taken into account to predict material hardness level.

Experiments presented in the paper showed that shear velocity yields more accurate hardness predictions than the longitudinal velocity. However, this can be an effect of lower accuracy in the measurements of TOF for the longitudinal wave, due to its higher velocity and the lower sampling frequency of electronic equipment used during the experiment. Moreover, measuring shear velocity using an EMAT is less complicated than the corresponding measurement for longitudinal waves, since it is a 'dry' measurement that does not require acoustical contact.

A disadvantage of the presented technique is that it requires precise measurement of material thickness. In the course of our experiment it could be precisely measured for all samples, which is rather difficult to accomplish during an in-line production process. This problem might be solved by using a more sophisticated set-up consisting of two transducers separated from each other by a known distance and measuring the TOF of ultrasonic surface wave between them.

Summarising, non-destructive ultrasonic methods can form a very useful tool in continuous monitoring of steel hardness during the production process.

6. Acknowledgement

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