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New Method of Determining Carbon Content in Steel as the Main Alloying Element

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Abstract- Traditional methods of determining the carbon content in steel as a major alloying element, are usually implemented in several methods; Like chemical analysis, atomic absorption, or the most common method is x-ray spectre analysis, where all elements in steel in addition to carbon can be quantified. These methods are rather expensive and there are only a limited number of institutions available. In some cases, an approximate estimate of the carbon content in the steel is needed only, and this can be implemented through the method of metallurgy, where the annealed steel samples are microscopically examined and from an estimate of the percentage of micro-components, an approximate percentage of the carbon content can be determined. All of the above techniques are destructive methods, required sample cutting, and preparation. In some cases these methods cannot be used, either because a rapid estimation is required and / or the testing must be performed on actual steel components, i.e. non-destructive inspection. An obvious option is ultrasound technology. Recently a research program was conducted on selected samples of normal carbon steel to determine the carbon contents in both annealed and conformal conditions, i.e. equilibrium conditions (fine equilibrium components of ferrite + perlite, perlite and perlite + cementite). The approach used was to try to take advantage of the expected variation in the ultrasound response of these micro-components in the steel, and thus correlate the carbon content with the ultrasound resonance amplitude using a pressure wave scanning technique. The results showed a linear relationship between the ultrasound echo amplitude and the carbon content. For the thick annealed steel section, a higher lower resonance amplitude as well as a higher attenuation rate of the reverberation amplitude compared to the normal case was indicated. It has also been concluded that the steel grain size and pearlite lamination appear to have little effect, and the higher test frequency (4MHz) in annealed perlite steels appears to reduce the chance of restoring good back wall resonance signals.Keywords- First

Keywords- *Non-destructive Examination, Ultrasonic Technique, Carbon steel, Annealed Pearlite, Ferrite*

I. INTRODUCTION

Non-destructive testing (NDT) techniques in the evaluation of material structure and prediction of material behavior began to evolve some thirty years ago [1]. The techniques included x-ray, non-destructive eddy current, neutron scattering, electromagnetic and ultrasonic Kimetal, have worked on the microstructure evaluation of a ferritic stainless steel by neutron scattering [2, 3]. Houska characterized the composition and structure by x-ray diffraction and polonschutz estimated the marten site content in steel by an electromagnetic method [4,5]. A new XRD technique was described to separately measure tetragonal ratio and the volume fraction of co-existing pate and lathe martensite of ultra-high strength steel and their different carbon content could be calculated [6].Glow discharge optical emission spectroscopy technique was used for carbon layer thickness determination of carbon steel (0.2%C) [7].Carbon content can be determined in a moment by a spark testing, which is an inexpensive and effective way of classifying the steel in an industry. Spark computer recognition was developed to determine the content of carbon for the purpose of increasing efficiency and reliability [8]. Other non-destructive be evaluation methods are often based on the consideration of physical mechanisms which control material behavior is specific ways. Among these, the ultrasonic technique is very useful and has been applied rather extensively. In addition to the determination of material parameters such as elastic modulus and densities [9]. The ultrasonic approach is based on how ultrasound waves are influenced when they are propagated within the material being tested. These waves undergo changes, which can be measured, and the material can be evaluated accordingly. The evaluation of the properties of the material can then follow indirectly, by means of graphical or empirical correlation. Hence, one can interpret certain changes in the ultrasonic signal as a change in the structure of the material [10, 11] has pointed out that inhomogeneous materials containing multiphase structure at their grain boundaries which are treated as ultrasonic interfaces; the acoustic impedance may change abruptly due to differences in acoustic velocity and/or density (acoustic impedance = velocity ×density) of the adjacent grains. This is applied to the structure of steels where either ferrite + pearlite, pearlite or cementite + pearlite are the micro constituents in the annealed or normalized condition, i.e. inhomogeneous multiphase structure which is found to differ in their response to ultrasound energy propagation.[12] Determination of elements by ICP-OES method. [13] This work examined the effect of carbon content on the microstructure and mechanical properties of DP steels. The carbon content of martensitic phase, grain size of martensite and ferrite, and the strength of both phases [14-16] strongly affected by the chemical composition of the steel. Many models were proposed to explain the behavior of DP steels [17]. Many authors explain the effect of the tempering temperature on the mechanical properties by the presence of reformed austenite [18-21] showed that (13%Cr4%NiMo) having a higher percentage of reformed austenite have a higher Charpy impact energy.

The present work is an attempt to apply the ultrasonic technique as a non- destructive method for estimating the carbon content in steels. Such a method must be quick, easy to carry out and evaluate and not too expensive. Basically, the principle involved in this method, was related the influence of different steel micro constitutes on ultrasonic echo amplitude with the carbon contents of the steel both graphically and by empirical equations.

II. MATERIALS AND EXPERIMENTAL TECHNIQUES

The materials used in this investigation involve a number of plain - carbon steels having different known carbon contents, covering; dead mild steel, mild steel, medium carbon steel, high carbon steel and tool steel, i.e. the most common classes of plain-carbon steels as indicated in Table 1.

TABLE I. PLAIN-CARBON STEELS INVOLVED IN THE AS RECEIVED CONDITION

Steel No.	Estimated % carbon
1	0.1-0.15
2	0.3-0.35
3	0.35-0.4
4	0.5-0.55
5	0.65-0.7
6	1.1-1.1

III. PRELIMINARY TEST

In order to have suitable size ultrasonic testing samples with different carbon contents covering most common plain-carbon steel cases, a large number of steel samples of 1/2 × 4 × 8 " (12.5 × 50 × 100 mm) in size were collected in addition to small representative specimens for metallographic purpose. After usual metallographic preparation, an approximate estimation of the carbon content of each specimen was then determined, depending on the % estimation of steel micro-constitutes in the as-received condition. Six specimens were finally chosen. Specimen having similar carbon content or possible non-equilibrium structure or not well defined structures were eliminated.

IV. ULTRASONIC TESTING SAMPLES PREPARATION

Six testing samples representing the steel involved were cut and machined to 12.5 × 50 × 100 mm dimensions. In order to have back wall reflections from different sound travelling distance (thickness) using normal (compression) wave probes scanning technique, number of steps were made in these samples to represent 10, 20, 30, 40 and 50 mm scanning distances as illustrated in figure 1. Both scanned surfaces and the reflecting surfaces were fine ground by grinding machine. The back wall surfaces have been treated as artificial reflectors in the steel testing samples. Hence the influence of steel thickness on the carbon content estimation can be demonstrated.

V. HEAT – TREATMENTS

Ultrasonic steel testing samples having different carbon contents with their small representative metallographic specimens were placed in a box-like steel container to avoid possible oxidation and carburization during heat treatment. Heat treatments involved were full annealing and normalizing their detail are shown in table 2. Both annealing and normalizing have been chosen mainly to study the influence of the equilibrium micro constituents in steels (ferrite + pearlite, pearlite + cementite + pearlite) on the ultrasonic response which are directly related to the carbon contents of the steel. In addition, the influence of steel micro constituent grain size and pearlite lamination are demonstrated using this technique.

TABLE II. DETAILS OF THE STEEL SAMPLES INVOLVED AND HEAT TREATMENTS

Steel No.	% Estimated microcontituents		Determined approx.% Carbon (ann. Cond.)	Annealing (F.C)	Normalizing (A.C)
	Ferrite	pearlite		Aust. temp. °c	Aust. temp. °c
1	87.5	12.5	0.1	940	940
2	62.5	37-5	0.3	880	880
3	50	50	0.4	880	880
4	37.5	62.5	0.5	870	870
5	12.5	87.5	0.7	810	810
6	cementite 5.2	94.5	1.1	760	880

VI. METALLOGRAPHIC

The metallographic steel specimens representing each ultrasonic testing samples were prepared for microscopic examination using the usual method of grinding, polishing and then etching by 5% Nital. The annealed specimens were carefully microscopically examined using ordinary metallurgical microscope. The revealed micro-constituent estimated percentages of these annealed steel samples were then determined as presented in Table 2. From the estimated values of various steel micro constituents, the approximate carbon content has been determined applying the Lever role principle and the steel portion chart. Representative photomicrograph of each steel involved are shown in figure 2 and figure 3 for both annealed and normalized conditions.

$\boldsymbol{Note}$: $30\ min. Soaking\ Period\ for\ both\ annealing\ and\ normalizing$:

$F.C = Furnace\ Cooling,$

$A.C = Air\ Cooling$

$Ann.Cond = annealed\ condition$

VII. ULTRASONIC TESTING

USM2 Ultrasonic flaw detector and probes of kraut Kramer, were used during this investigation. Screen calibration was done using the VI-reference block with suitable testing sensitivity level (No. of dB required to bring the maximum echo amplitude to a certain height of the oscilloscope screen) 80% F.S.H. (Full Screen Height) was found adequate throughout the whole range of thickness, using SAE30 engine oil as a complaint. The probes were set to obtain the maximum echo height possible from the back wall surface of the testing samples steps by moving the probe forward and backward and by rotation i.e. maximum echo amplitude technique, as illustrated in Fig 1.

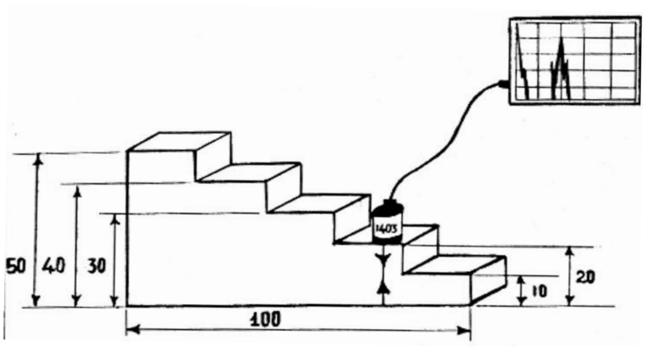

Figure 1. Design dimension of the ultrasonic testing sample, illustrating the normal probe scanning.

Ultrasonic variables such as testing frequency and scanning distance were varied in order to establish an effective combination of these parameters for testing the condition of the steels involved in both annealed and normalized conditions. Hence the influence of metallurgical factors such as grain size and pearlite lamination can also be studied. The aim from varying heat-treatment conditions is to establish which of the two conditions is more applicable by using this technique.

VIII. RESULTS AND DISCUSSION

The results obtained in this article are trying to directly relate the variation in the ultrasonic response with the equilibrium microstructure of steel. This has been treated as the main controlling factor for estimating carbon contents in both annealed and normalized steels.Plain-carbon steels equilibrium micro-constituents are ferrite, pearlite and cementite. The type of constituents and the relative percentage depend mainly on the carbon content of the steel, while grain size is related to the heat-treatment condition. As the carbon content was increased in the hypo eutectoid steel range, the percent of pearlite will increase, reaching 100% at 0.8% carbon steel i.e. eutectoid steel, while ferrite relative percent will decrease. In the hypereutectoid steel range, an intermetallic cementite constituent will appear as a pearlite grain bounding net-work, it's percent starts to increase with increasing carbon content. This has been clearly demonstrated in the micrographs of Figs. 2 and 3.

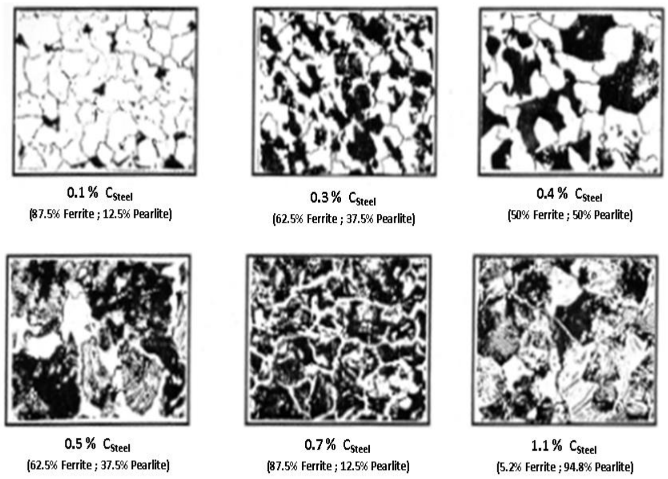

Figure 2. Micrographs representing various carbon steel samples involved in the annealed condition X32.

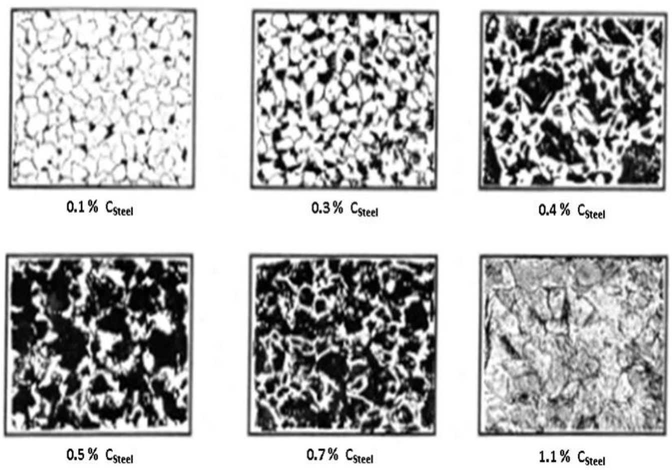

Figure 3. Micrographs representing various carbon steel samples involved in the normalized condition X320.

Hence dealing with steels ultrasonically, means multiphase structure materials are involved. It is, therefore, not surprising that then- acoustic properties are also fluctuated within a wide range. This has been shown as fluctuations in the ultrasonic echo amplitude height (signal's strength) and their attenuation rates, which appeared to be due to the steel's micro constituents different behavior towards ultrasonic waves as illustrated in Figs. 4 – 7, hence indirectly relating carbon content.

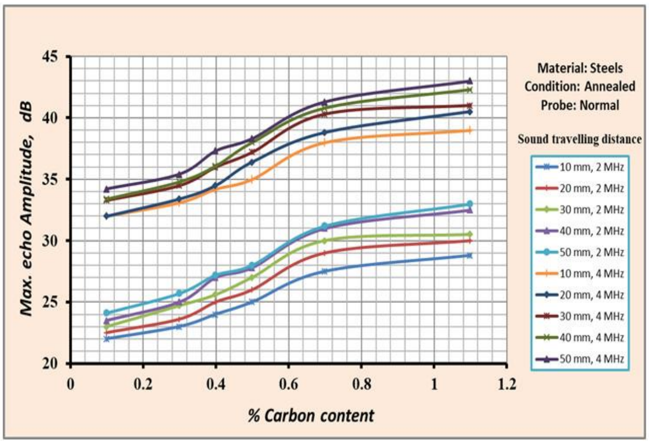

Figure 4. Correlation between Ultrasonic maximum echo amplitude with % carbon content of annealed plain carbon. steel samples, using both 2MHz and 4MHz frequencies compression probes.

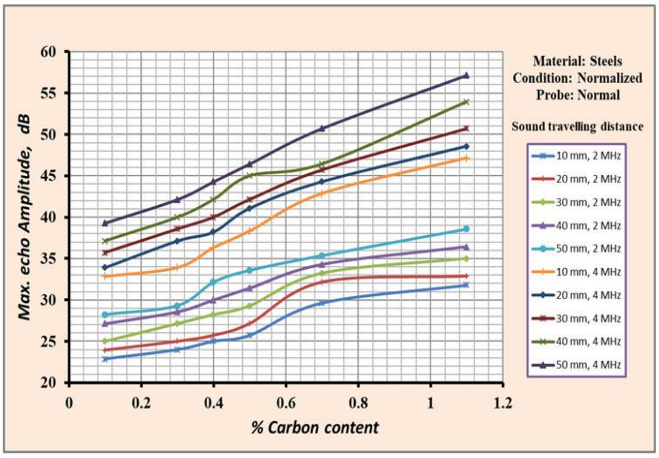

Figure 5. Correlation between Ultrasonic maximum echo amplitude with % carbon content of normalized plain carbon steel samples, using both 2MHz and 4MHz frequencies compression probes.

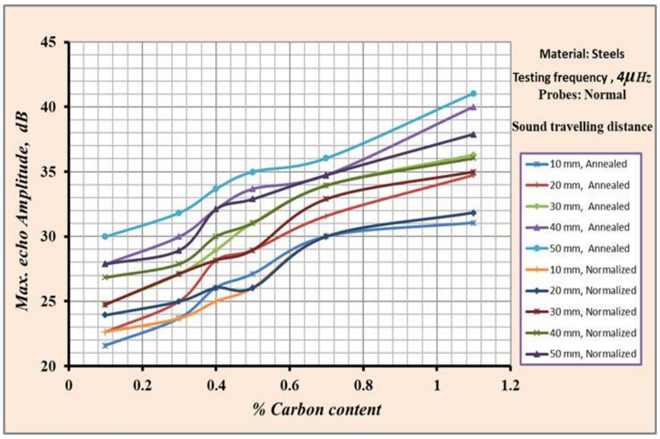

Figure 6. Comparison between annealed and normalized plain carbon steel samples, when carbon content is to estimated ultrasonically using 2MHz frequency compression probes.

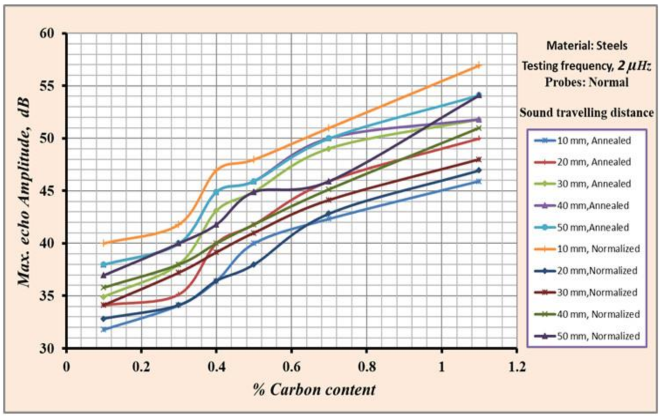

Figure 7. Comparison between annealed and normalized plain carbon steel samples, when carbon content is to estimated ultrasonically using 4 MHz frequency compression probes.

As stated earlier in the introduction of various ideas that have been suggested by a number of investigators [16,18,19,20,25], they have tried to explain how ultrasonic waves behave with multiphase structure materials such as plain - carbon steel, in terms of ultrasonic energy loss mechanisms. To reduce the amount of experimental work, it was decided to limit this study to the various micro constituent combination in different carbon contents steel structures samples in the annealed and normalized conditions only i.e. equilibrium heat treatments, in order to be able to vary grain size and pearlite lamination in the steels matrices involved, hence establishing their influence on the ultrasonic echo amplitude response i.e. metallurgical factors affecting this technique. A linear relationship seemed to exist between the carbon content of steels and the echo amplitude height, as clearly demonstrated in both figure 4 and figure 5. The results in these two figures represent the overall ultrasonic response of the steel involved in this investigation, at both 2 MHz and 4 MHz testing frequencies, using normal probes.

To simplify the discussion, it was decided that, the results are to be treated with reference to the two major direct influencing factors which have been involved during this investigation.

IX. ULTRASONIC VARIABLES

The results in Figs. 4 and 5 indicate that, as the carbon content increases, steel thickness (scanning distance) becomes critical, especially when 4 MHz probes are used. This is clearly illustrated in the case of tool steel (1.1% steel) at 50 mm thickness. The results indicate that relatively both high carbon content and/or thickness section can cause ultrasonic inspection problems during carbon content estimation, i.e. thickness parameter is to be seriously taken into consideration. Both Figs. 4 and 5 clearly illustrate this important variable and the technique employed appeared to have the ability for good resolution within the thickness values range involved. For reliable carbon content estimation, the results obtained in this investigation, strongly recommend that the knowledge of both thickness and approximate carbon content range are essential before applying charts of this nature, especially for high carbon steels and tool steels of more than 2 inches (50 mm) thick applying 4 MHz frequency probes. The influence of steel thickness is clearly demonstrated in both annealed and normalized states using both 2 MHz and 4 MHz probes. The results also illustrate the influence of testing frequency and the 2 MHz frequency probes are highly recommended for their relatively high echo amplitude response and low rate of amplitude attenuation. This is appeared to agree with a number of investigators [22-24].

The ultrasonic variables which are involved in this investigation such as scanning distance and testing frequency have shown a combined effect of sound penetration restriction (thickness limitation) and high amplitude decay i.e. high testing frequency (4 MHz) reduces the chance of recovering good back-wall echo signals specially when thick steel sections are involved. This is thought to be related to the increasing in the energy loss of ultrasound wave due to both attenuation and beam divergence phenomena as the penetration depth increases [22, 24].

X. METALLURGICAL VARIABLES

The results obtained which are illustrated in Figs. 4 and 5 demonstrate the general low echo amplitude height and high echo amplitude attenuation rate as the carbon content of the steel is increased. This can be related to the increase percent of relatively attenuated 2-phase pearlite structure in both annealed and normalized steel structures. The results confirm those obtained by Lee[1] and Smith[12], as they studied the effects of steel, carbon content metallurgical state and gain size on ultrasonic attenuation in steel. Their results showed that grain type in steel and their relative percentages can be influenced by the chemical composition, mainly by carbon content, while grain size is greatly affected by heat-treatment and both have led to high attenuation. The various steel micro constituents have been found to exhibit different ultrasonic attenuation for the same size [1,20,23-26]. Szilandin [21] in his investigation has found that ultrasonic energy loss due to scattering does not only depends on grain size, but also on the grains kind. His results confirm those obtained in this investigation when comparing the ultrasonic response of annealed microstituents with those of normalized steels as illustrated in both Figs. 6 and 7. The normalized steels curves show similar characteristics to those of annealed steel samples, but foe actual values have changed. The results indicated slightly lower echo amplitude height and higher echo amplitude attenuation rate in the annealed steel, especially when relatively thick sections are involved. This is thought to be related to the larger grain size and coarser pearlite lamination in the annealed structure. The micrographs of Figs. 2 and 3 have confirmed this variation in the microstructure of plain - carbon steels in the two different heat - treatments involved. Both Figs. 6 and 7 were constructed mainly to demonstrate foe influence of heat - treatment conditions (annealing and normalizing) of the steels using this technique, but their results do show that in general, there is not so much different in foe ultrasonic response whether the steel is annealed or normalized within the grain size difference and the steel thickness range involved.

Although there has been a clear indication in both Figs. 6 and 7 that in relatively thick sections for steel containing high percentage of pearlite i.e. high carbon steel or tool steel; the difference in ultrasonic response between annealed and normalized steel has to be taken into consideration.

XI. Empirical Formula for Carbon Content Estimation in Plain-Carbon Steel

The graphical presentation of carbon contents of steel related to the echo amplitude height, which have been dealt with earlier, can also be illustrated in the form of empirical formulae as mathematical presentation by selecting the best curve fit result in each case with confidence limit of 95%. The following equations can be used to represent the relationship between ultrasonic echo amplitude in dB and steels carbon contents, using 2MHz and 4MHz testing frequencies normal probes and the 50 mm* sound travelling distance (thickness) has been chosen.

Annealed steels:

% C= 0.093 * Max. Echo Amplitude in dB-2.75 using 2MHz

% C= 0.057 * Max. Echo Amplitude in dB-2.22 using 4MHz

Normalized steels:

% c = 0.093 * Max. Echo Amplitude in dB-2.6 using 2MHz

% c = 0.053 * Max. Echo Amplitude in dB-1.9 using 4MHz

XII. Evaluation of the Technique Capability

The results obtained during this investigation demonstrate the capability of the ultrasonic technique employed to correlate echo amplitude response with carbon content of steels, by foe conversional maximum echo amplitude method using compression (normal) probes scanning. Both graphical correlation and mathematical correlation which is involved in this investigation can be used to estimate carbon content in steel non-destructively. Although the number of results taken are limited. Note, for other thickness values, modified equations are to be used. But they can to be considered as indicative of positive correlation. It should also be emphasized, that such correlation would not be as simple as indicated in this investigation as a linear correlation. This is because factors which can influence the microstructure were eventually found to affect carbon content estimation.

XIII. Conclusions

The results obtained in this investigation, can justify the following conclusions:

1. Linear relationship seemed to exist between ultrasonic echo amplitude and carbon content in plain-carbon steel.

2. General low echo amplitude height and high echo amplitude attenuation rate were shown, as the carbon content of the steel was increased.

3. Steel thickness becomes critical as the carbon content was increased, especially when high testing frequency (4 MHz) probes were used.

4. Slight indication of lower echo amplitude height and higher echo amplitude attenuation rate, especially when relatively thick steel sections were involved, if the steel sampleswere annealed as compared with normalized condition.

5. Grain size & pearlite lamination in steel samples appeared to have little effect, but the type of grains and their relative percentages appeared to be the controlling factor i.e. elastic mismatch between the adjacent grains.

6. High testing frequency (4 MHz), reduced the chance of recovering good back wall echo signals in annealed pearlite steel (>80% pearlite)

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