TESTING METHODS FOR HEAT TREATED STEEL PRODUCTS

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1.0 INTRODUCTION:

Heat treatment of steels changes the microstructure. For the same chemical composition of a steel, the microstructural variations is the most important parameter that determines the properties of a component - physical, chemical, mechanical and others. The presence of a wrong or undesirable microstructure reduces the life of a component and often leads to premature failures. Another parameter which is important for component integrity is the presence of physical discontinuity such as cracks which are introduced in the component due to faulty heat treatment which imparts wrong microstructures and residual stresses. The microstructural features have a wide range of dimensions. Figure 1 gives an idea of the dimensions of various microstructural features. The types of testing methods chosen would depend on the particular types of microstructural features one is interested in.

A testing method can be either "non-destructive" (i.e. tests carried out on the heat treated components), or they can be carried out on small specimens heat treated along with the actual components. The choice and use of either or both of these two categories of techniques depends on the techniques that are available, the size of the component, and the total area to be covered by the testing method. The testing methods can be classified in two categories in another sense: (a). Techniques that measure parameters related to microstructure such as hardness, mechanical strengths, and residual stress, or based on microstructures such as grain size, and relative amounts of phases, (b). Techniques that detect and assess defects in the form of physical discontinuities such as macro cracks.

This paper considers various testing methods including those conventionally used and those which have potential for use.

2.0 HARDNESS MEASUREMENT

2.1. Conventional Techniques of Measurement:

The term "hardness" usually implies a resistance to plastic deformation. Conventionally, there are three types of hardness measurements : (a) scratch hardness of interest to mineralogists, (b) rebound technique in which the height of rebound of an indenter dropped on a metallic surface is considered as a criterion of hardness, and (c) indentation hardness which is related to the depth of penetration of a suitable indenter into a metallic material when a suitable load is applied to the indenter for a predetermined time and then released. The last mentioned technique is the most popular and relevant for a metallic material like steel. Depending on the type of the indenter, there are several variations of the hardness testing. They are given in the following table :

Table 1 : Indenter and Load Particulars in Various Hardness Measurement Schemes

Hardness type	Indenter type	Load
Brinell	10mm dia steel ball Tungsten ball for very hard material	500 kg for soft material 3000kg for hard material
Vickers	Square based diamond pyramid with 136^o as the included angle between the diagonals	1 to 120kg.
Rockwell	20^0 diamond cone with a slightly rounded point, and 1/16 inch and 1/8 inch steel balls	60, 100 and 150kg.
Micro-hardness Vickers	Square based diamond pyramid	usually 50 -100 g. for steel
Micro-hardness Knoop	Diamond ground to a pyramid form with the long and short diagonals in the ratio of 1:7	usually 50 - 100 g for steel

The following equations are used for expressing the hardness numbers:
Brinell Hardness Number:

$$BHN = \text{Load/Surface area of indentation}$$
$$= P/[\pi D/2\{D - (D^2 - d^2)^{1/2}\}] \qquad (1)$$

Where P = applied load in kg; D = diameter of ball, mm,
d = diameter of indentation mm; t = depth of impression, mm

BHN is not a satisfactory physical concept since equation (1) does not give the mean pressure over the surface of the indentation. BHN generally varies with load. Therefore it is not possible to cover with a single load the entire range of hardness encountered in metallic materials. The relatively large size of Brinell impression is an advantage in averaging out local heterogeneity.

Meyer Hardness:

$$MH = 4P/\pi d^2 \qquad (2)$$

where d= diameter of the projected circle of the indentation. MH is then a more rational definition of hardness than BHN since a mean pressure P is implied over the projected area. It is therefore less sensitive a function with applied load.

Vicker's Hardness (VHN) also known as Diamond Pyramid Hardness (DPH):

$$VHN = [2P. \sin(\theta/2)]/L^2 = 1.854P/L^2 \qquad (3)$$

Where P = applied load, kg; L = averaging length of diagonal, mm
θ = angle between opposite faces of the diamond = 136^o

Vicker's hardness test has received wide acceptance for research work because, it provides a continuous scale of hardness for a given load from very soft metals with a VHN of 5 to hard materials with VHN more than 500. With Rockwell or Brinell hardness test, it is usually necessary to change either the load or the indenter at some point in the hardness scale, so that measurements at one extreme of the scale cannot be strictly compared with those at the other end because the impressions made by the pyramid indenter are generally similar no matter what their size, VHN is independent of load except at very light loads.

Rockwell Hardness Test

It is a very popular test technique. Its general acceptance is due to its speed, ability to distinguish small hardness differences in hardened steel and the small size of the indentation, so that finished heat treated parts can be tested without damage. The test utilises the depth of indentation, under constant load, as a measure of hardness. A minor load of 10kg is first applied to preset the specimen. This minimises the amount of surface preparation needed and reduces the tendency for ridging or sinking in by the indenter. The major load is then applied, and the depth of indents is automatically recorded on a dial gauge in terms of arbitrary hardness numbers.

2.2. MICRO HARDNESS TESTS:

Measurement of the hardness gradient at a carburised surface the determination of the hardness of individual constituents in a microstructure or the checking of the hardness of a delicate watch gear are typical examples of micro hardness measurements. The special shape of the knoop indenter makes it possible to place indentations much closure together than with a square Vicker's indentation e.g. to measure a steep hardness gradient. Besides, the volume indented by a Knoop indenter is much less than those made by Vicker's indenter. This is useful when measuring the hardness of a thin layer.

2.3. HARDNESS-CONVERSION RELATIONSHIPS :

Relationships of hardness numbers of one type to another are empirical in nature. The most reliable hardness conversion data exist for steel harder than 240 Brinell. ASTM, ASM, and SAE have agreed on a table for conversion between Rockwell, Brinell and VHN which is applicable to heat treated carbon and alloy steel and to almost all alloy constructional steels and tool steels in the as forged annealed, normalized and quench/tempered conditions. [ASTM Standard E-140-78. ASM Handbook, Vol. 4, 1991].

2.4. RELATIONSHIP BETWEEN HARDNESS AND FLOW CURVE :

Attempts have been made to understand the characteristics of deformation during indentation and thus able to assess the state of toughness and degradations if any in service exposed components. In some cases relationships have also been found between the hardness values and tensile strengths [Figures 2 and 3]. Relationships such as these have lent confidence in the use of hardness as a monitoring parameter for the study of the evolution of microstructures in variously heat treated steels.

2.5. OTHER TECHNIQUES OF THE MEASUREMENT OF HARDNESS :

While the standard techniques of measurement of Brinell, Vickers and Rockwell hardness as discussed above are conventional, they cannot always be used on heat treated components. Techniques are now available to measure hardness on actual components whatever may be their size and shape.

2.5.1. Magnetic Techniques Of Hardness Measurement:

It is often the case that a mechanically hard steel is also magnetically hard and vice versa. In simple terms, a magnetically hard steel means higher coercivity (Fig. 4). Therefore coercivity can be taken as a parameter as a measure of hardness. However, the accurate measurement of coercivity requires the availability of toroidal specimens [Fig. 5] and is time consuming. Also, if other techniques are to be considered, then they should have advantages over the conventional indentation techniques. One advantage could be in terms of the applicability as a non-destructive test technique. In this sense coercivity is not a suitable parameter though attempts are being made to use parameters which are equivalent to coercivity. Retentivity [Fig. 6] could be used as a NDT parameter, since for this it is only needed to measure the residual field by a Hall effect probe when a slowly varying magnetic field introduced through an yoke is switched off. However it has not been found to be a good parameter to act as an yardstick of hardness. It is therefore needed to consider magnetic Barkhausen noise (MBN) which has attracted recent attention for the measurement of residual stress, hardness and characterisation of microstructures. The origin of MBN can be related to the fine scale step like structure of the contour of a magnetic hysterisis loop that signify the motion of domain wall from one pinning point to another [Fig. 7]. Such domain wall motions create magnetic signals that can be sensed by a sensor coil. A typical block diagram of MBN instrumentation is shown in Fig. 8. The electromagnetic assembly shown in the figure corresponds to a laboratory setup. For inspection in a component "U" shaped yokes can be used to magnetise a small portion of surface of the component, and the sensor coil can be positioned between the two legs of the yoke. Commercial equipment are nowadays available for MBN acquisition and analysis. Since coercivity is a manifestation of the impediment against the movement of domain walls, a higher coercivity will mean lower MBN signal strength. Therefore, higher mechanical hardness will mean lower MBN signal strength and vice versa.

One of the common parameters in MBN analyses is the height of the peak of the rms voltage plot of MBN signals acquired over half of a magnetic hysterisis sweep [Fig.9]. The hardness gradient can be obtained by digital filtering since MBN signals are electromagnetic in nature and an eddy current effect is associated with such signals.

While the MBN technique is simple to use and not very expensive it is necessary that the effect of microstructural features and the effect of residual stresses on MBN should be understood. In some cases it has been found that MBN peak height and coercivity are not inversely related.

2.5.2. XRD Technique For Hardness Measurement:

Availability of portable X-ray diffraction units which are increasingly being used for the measurement of residual stresses, calls for an understanding as to whether such an equipment can also be used for the assessment of hardness in heat treated steels. In fact the FWHM (Full Width at Half Maximum) of X-ray diffraction peak obtained by a portable XRD equipment has been used to monitor the peak hardening behaviour of quench aged aluminum alloys. This has shown a possibility of correspondence with hardness. No systematic investigations have been carried out on the use of FWHM for the assessment of hardness in heat treated steel. Preliminary work carried out by the author on alloy steels has shown that in some cases of quenched and tempered steels this parameter can be used.

2.5.3. Ultrasonic Method For The Measurement Of Hardness:

Ultrasonic velocity is a function of the elastic modulus. The modulus is affected by the presence of internal stress and the presence of second phase precipitates. It is therefore, expected that ultrasonic velocity should be a parameter that can be correlated with hardness in practice. However, there are a few points that should be noted in such a case. The variation in the modulus and ultrasonic velocity due to the variations in microstructure effected by heat treatment is small and of the order of about 4-5% maximum. Therefore the accuracy needed for the measurement of ultrasonic velocity for such applications is very high. This is for this reason that the use of velocity is yet to find application to test heat treated steels.

Attenuation of ultrasonic waves passing through a heat treated product is another ultrasonic parameter that can be used to monitor the product. Attenuation depends in a complex manner on the microstructural features such as grain size, dislocation substructure, second phase precipitates, and also depends on the test frequency. The problem of theoretical understanding on the nature of the dependence is formidable. Therefore, empirical approach is normally undertaken in the investigations. Though ultrasonic attenuation is sometimes used to measure grain size in steels, and good correlation has been found between attenuation and fracture toughness in steels, it has so far not been used to monitor hardness.

2.5.4 "Equotip" Measurement Of Hardness:

This is a commercial equipment based on the principle of rebound of a suitable impact device. This is portable and gives fairly reproducible results.

3.0 GRAIN SIZE MEASUREMENT BY NON-DESTRUCTIVE MEANS

Grain size in the microstructures is an important parameter that affects mechanical properties, such as yield strength and impact energy. The following non-destructive test parameters can be used to measure grain size.

(a) Ultrasonic attenuation co-efficient
(b) Ultrasonic back scatter characteristics
(c) Coercivity and
(d) MBN peak height.

All these four parameters need further studies to establish under what conditions one or more of them can be used to measure grain size. It may, however, be noted that in a few cases, successes have been reported by using these parameters. Fig. 10 shows the variation of coercivity as a function of the inverse of prior austenitic grain size in 9Cr-1Mo steel water quenched from different austenitization temperature.

4.0 TEST METHODS FOR RESIDUAL STRESSES

The test methods which are applicable for the measurement of residual stresses in heat treated products are the following:

(a) X-ray diffraction based techniques
(b) Barkhausen noise analysis based technique
(c) Hole-drilling and trepanning technique used with strain gauge, and
(d) Ultrasonic velocity measurement based technique.

Out of these 4 techniques the first 3 are NDT techniques and can be used in actual components. Commercial systems are available for the first two. The third technique i.e., ultrasonic technique is still in the developmental stage. The fourth technique i.e., strain gauge technique needs hole-drilling or trepanning and may not be acceptable for use on a heat treated component. However, a large amount of information is available on this technique. Details of the techniques can be found in another presentation in this workshop devoted exclusively to residual stresses.

5.0 TEST METHODS FOR DETECTING AND ASSESSING DEFECTS IN THE FORM OF PHYSICAL DISCONTINUITIES

Perhaps the only type of defect in the form of a physical discontinuity that can form due to a heat treatment process is crack. The techniques which are in significant industrial use for crack detection and assessment are the following:

(a) Liquid penetrant testing (LPT) or dye penetrant testing (DPT)
(b) Magnetic particle testing or inspection (MPT or MPI)
(c) Eddy current testing
(d) Ultrasonic testing, and
(e) Radiography.

The LPT which depends on the entry of a penetrant chemical (fluorescent or otherwise) inside a surface breaking crack by capillary action, which is subsequently revealed by the action of a developer is a well known technique and is not discussed here in details. For ferromagnetic steels for which MPT is possible, the latter is often preferred against LPT because of higher speed of testing, higher sensitivity of crack detection in many cases, and non-necessity of surface cleaning.

Figure 11 shows the electromagnetic phenomena utilized in detecting surface breaking cracks. : (a) residual leakage field (region BC); (b) active field (region OA); and (c) ac excitation (region ABCDEA). The first two are applicable in MPI and the third in eddy current testing.

In MPI where the presence of a crack is revealed by the accumulation of magnetic powder (dry or wet fluorescent) attracted by the leakage flux, the choice of type of magnetisation (ac or dc), active leakage flux or residual leakage flux etc. depends on the material, crack depth, whether the crack is surface breaking or sub-surface etc. The eddy current testing which depends on the monitoring of the change in the magnetic flux pattern due to the occurrence of eddy current owing to the presence of a crack is not applicable for ferromagnetic steels because of the fact that the variation in the magnetic flux pattern depends strongly on the localised variation of magnetic permeability. Therefore, it is mostly not possible to distinguish the signals due to the presence of defects and due to the variation of magnetic permeability. However, for non-ferromagnetic steels and nonferrous alloys eddy current testing is a very sensitive technique for the detection of cracks.

In ultrasonic crack detection a suitable ultrasonic wave is sent into the component by a suitable ultrasonic wave generator. For crack detection, either a shear wave or a surface wave is chosen. Conventionally they are generated by piezoelectric and magnetostrictive transducers. Presently, however, newer techniques such as EMAT (electromagnetic acoustic transducers) and laser induced ultrasonics are also possible. The ultrasonic waves either reflected from the crack surfaces or diffracted from the crack tips can be detected by a piezoelectric transducer, magnetostrictive transducer or a EMAT. Generally speaking i.e. if all types of metallic materials are considered, then ultrasonic crack detection is the most popular. The maximum amount of R&D efforts are also being put for this technique. However, for a ferromagnetic steel, if one considers the level of operator skill and intelligence needed, the speed of testing and even sensitivity and confidence of detection, then in many cases, MPI is considered a better choice for the detection of surface breaking cracks.

Radiography is essentially a shadow graph of X-Rays or gamma rays passing through a component. The X-rays are nowadays generated in a very wide energy range (a few tens of kV to 30 MeV) in order to be able to penetrate a wide range of component thicknesses. The energy of the gamma rays is, on the other hand limited since gamma rays of importance to radiography are generated only by a few radioactive nuclei such as Iridium-192 (Energy: 296 to 470keV), Cesium-137 (Energy: 662keV), and Cobalt-60 (Energy: 1.17 and 1.33MeV). The conventional means of the detection of the X-rays or the gamma rays after the passage through a component is by using films. However, nowadays solid state detectors aided by digital image processing is able to give Radiographic images of similar quality as obtainable by film radiography. Radiography is not a very suitable method for crack detection since the detectability depends on the orientation of the direction of the interrogating radiation with respect to the direction of the cracks (the best detectability occurring when the relative orientations are parallel to each other), and also because of the high cost of equipment, and slowness of operation.

6.0 MICROSCOPY OF HEAT TREATED PRODUCTS:

The microstructure of a heat treated product which cannot be sacrificed can only be obtained by in-situ metallography. Otherwise, conventional laboratory metallography can be carried out by cutting a small specimen from the component. In-situ metallography is nowadays a well established technique. Various types of commercial equipment for in-situ grinding, polishing, and microscopic observations (up to a magnification of 400) are available. For observations above this magnification a replica obtained from an in-situ etched surface can be examined by a scanning electron microscope. The use of in-situ metallography assumes that even the small amounts of polishing/grinding on the surface would not affect the performance of the heat treated product.

7.0 CONCLUSIONS:

The previous discussions give only a very brief account of the test methods that can be used for a heat treated product. It is realized that a great length of discussion is not possible because of space restrictions. Therefore, the discussions have neither been comprehensive nor in-depth. The author will be too willing and happy to share any more information that he would have regarding the test methods with the participants of the workshop.

FIGURE CAPTIONS

Fig. 1. Dimension Ranges of various Microstructural Features

Fig. 2. Relationship between Tensile Strength and Hardness for Quenched and Tempered, Annealed, and Normalized Steels. Ref: p-320 of "Mechanical Metallurgy" by G.E.Dieter 3rd Edition. McGraw Hill Book Company. 1986.

Fig. 3. Relationships between Tensile Properties of Quenched and Tempered Low-Alloy Steels. Ref: Same as for Fig. 2. p - 321.

Fig. 4. Linear Relationship between Coercivity and Hardness of Pearlitic Rail Steels. Ref: Bussiere J.B., "Applications of NDE to the Processing of Metals", in Review of Progress in Quantitative Non-Destructive Evaluations", Vol. 6B. Plenum Press. 1987.

Fig. 5. Circuits for Automatic Reading of B-H Curves and Loops. The Windings for Applied Magnetic Field and for Sensing of Induced Magnetic Field are done around the Toroid Specimen shown.

Fig. 6. B-H and M-H Magnetic Hysteresis Loops. Point D Corresponds to the Coercivity Point. OD is a measure of Coercivity. Point C Corresponds to the Retentivity. OC is a measure of Retentivity.

Fig. 7. Hysteresis Loop with a Schematic Enlargement of a Portion as Shown in the Inset. The Steplike Structure of the Contour Signifies Jumping of Domain Wall from one Pinning Point to Another giving rise to Voltage Spikes in a Sensor Coil

Fig. 8. Set up for MBN Signal Acquisition and Analysis.

Fig. 9. B-H Loop and MBN rms Voltage Plot for a Water Quenched 17-4PH Stainless Steel.

Fig. 10. Variation of Coercivity in Water Quenched 9Cr-1Mo Steel as an Inverse Function of Prior Austenite Grain Size.

Fig. 11. Electromagnetic Phenomena utilized in Detecting Defects in Ferromagnetic Materials: (a). Residual Leakage Field (Region BC); (b). Active Leakage Field (Region OA); and (c) ac Excitation (Region ABCDEA).

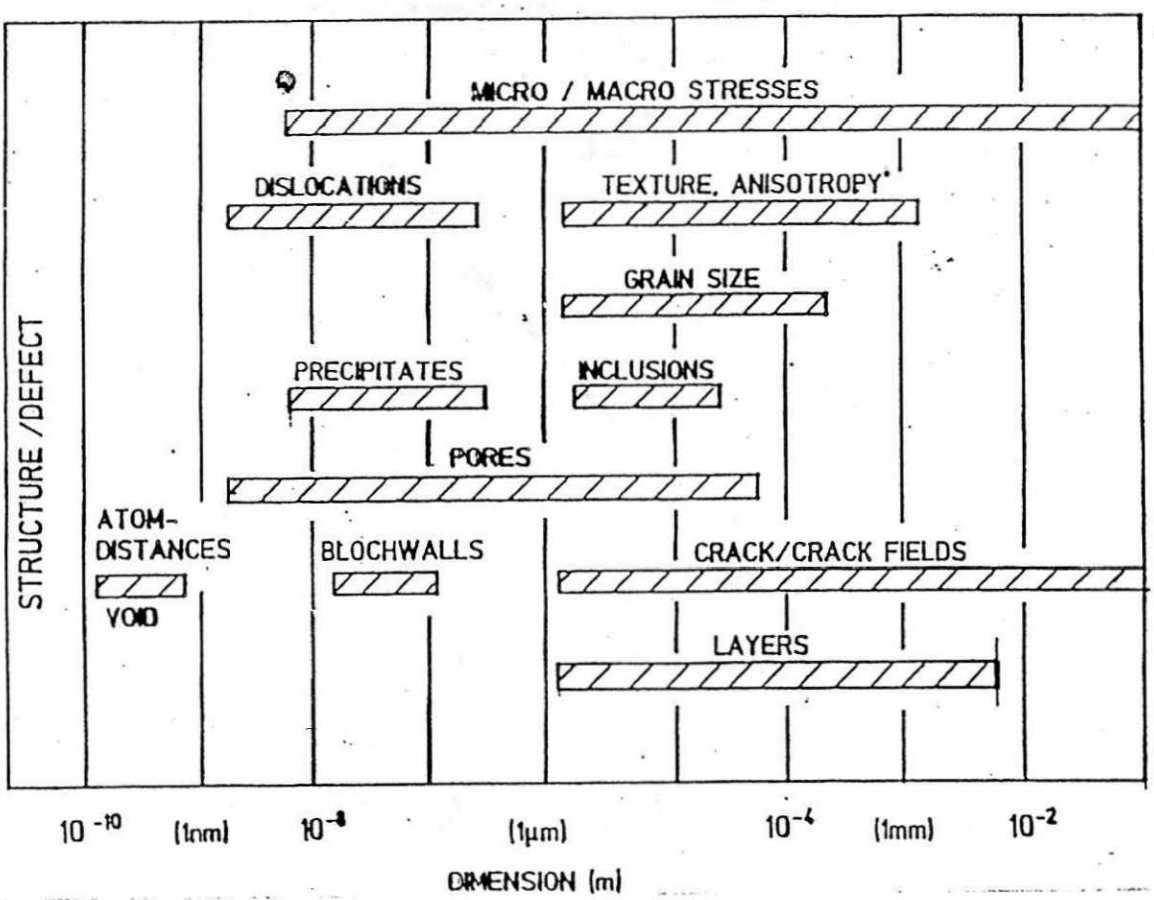
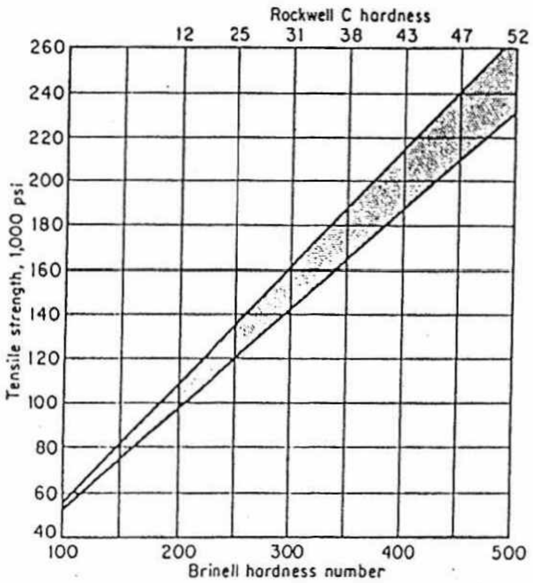


Fig.1: Dimensions of various microstructural Features and Defects

320 APPLICATIONS TO MATERIALS TESTING



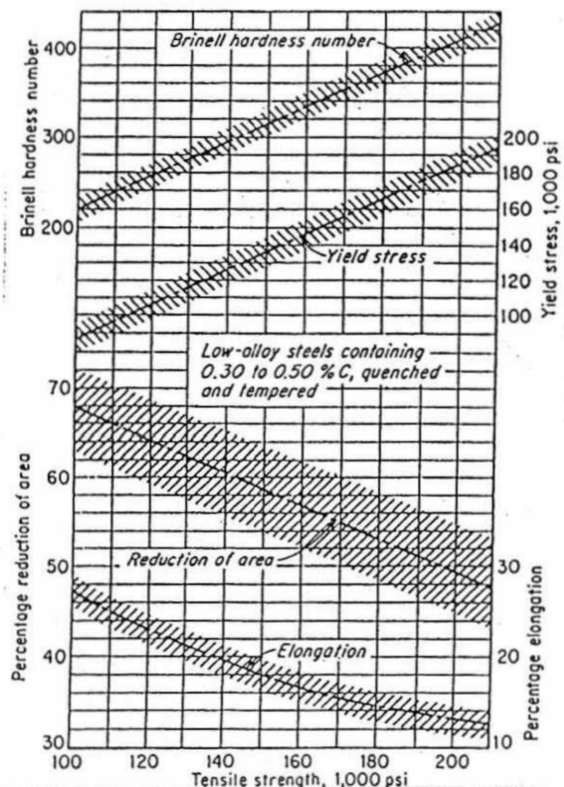


Fig.2: Relationship between tensile strength and hardness for quenched and tempered, annealed, and normalized steels *(From SAE Handbook)*

Fig.3 : Relationship between tensile properties of quenched and tempered low-alloy steels (From W.G.Patton, Met. Progr. Vol. 43.p. 726, 1943)

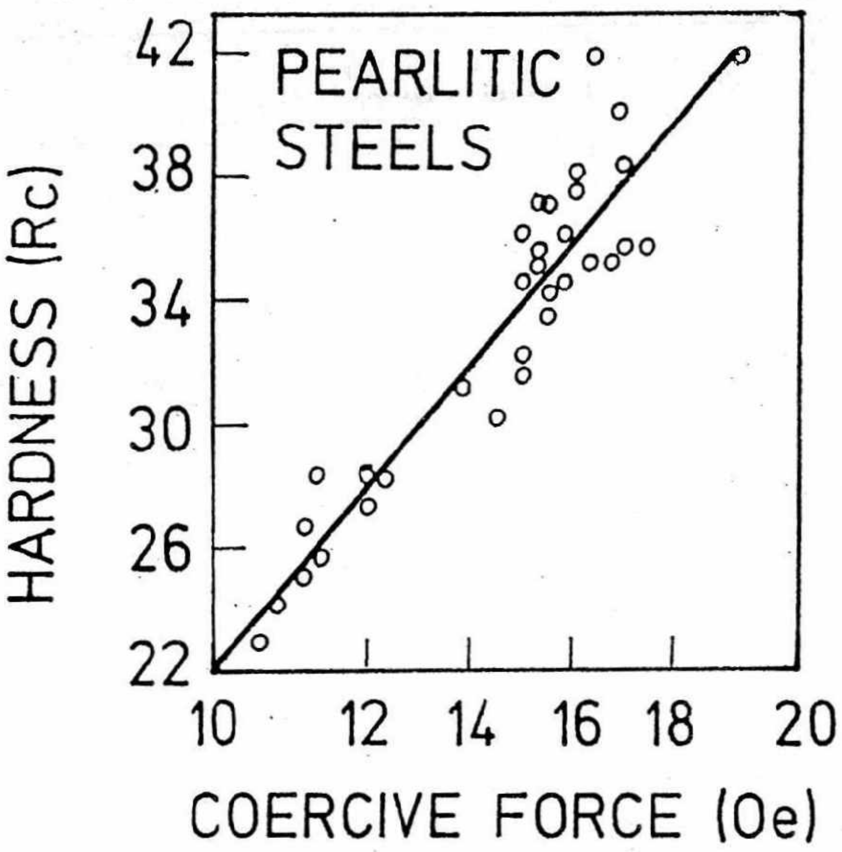


Fig.4:

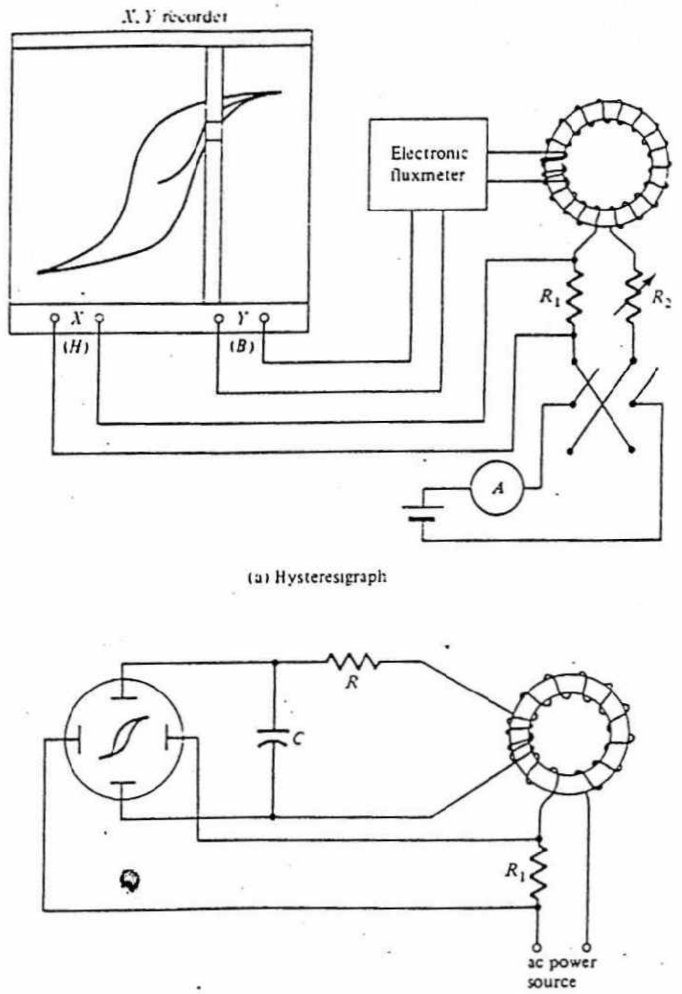


Fig. 5:

(a) Hysteresigraph

(b) Loop tracer

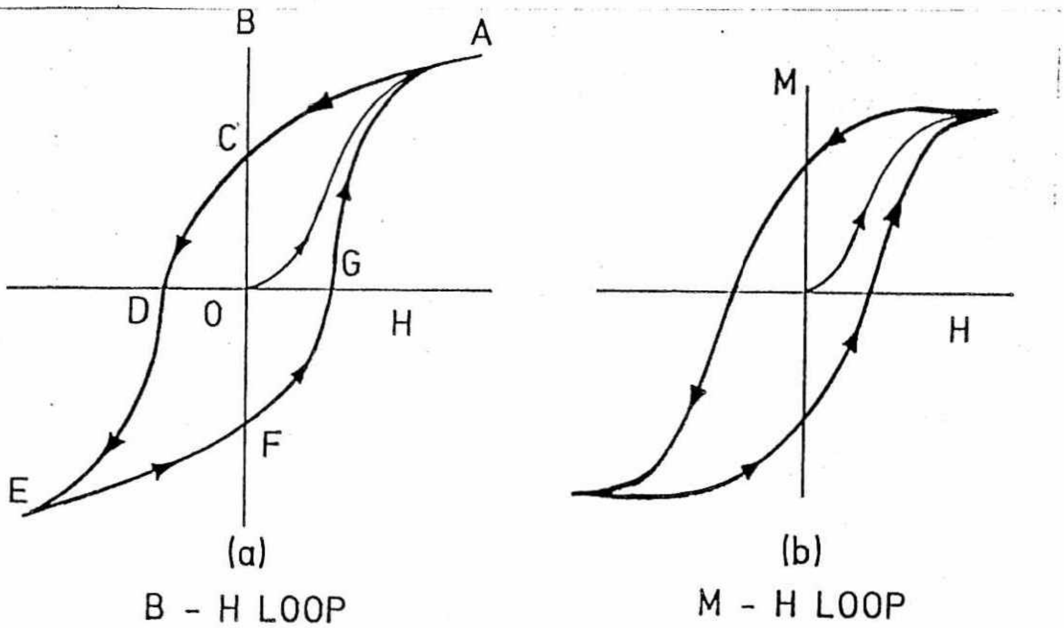


Fig.6:

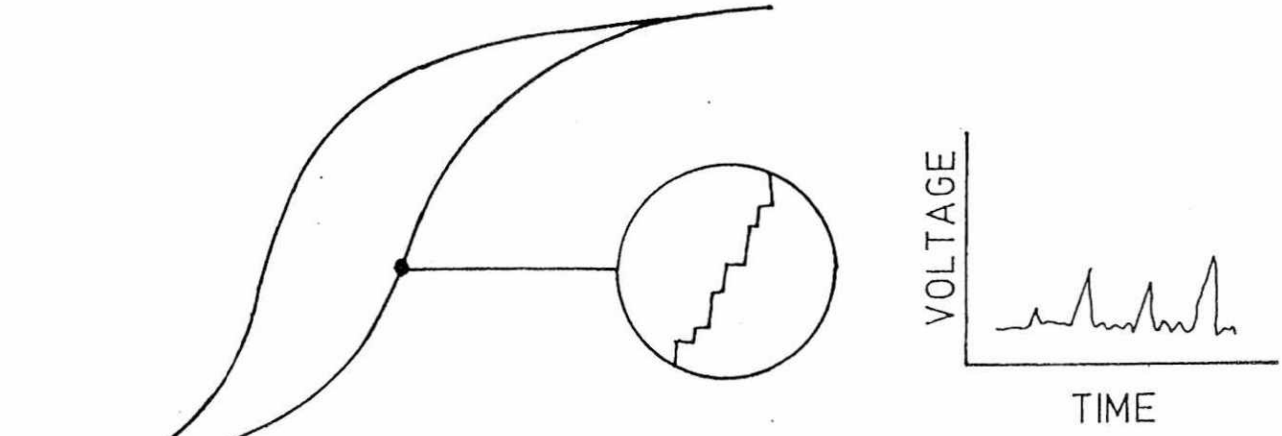
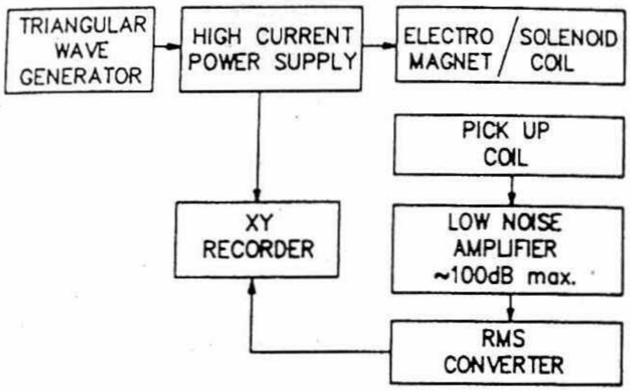
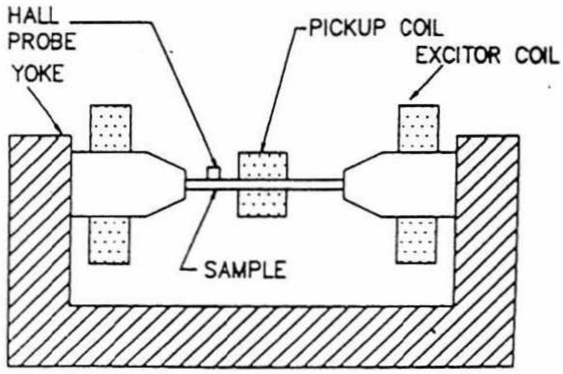


Fig.7:



BLOCK DIAGRAM OF SETUP

Fig.8:



ELECTRO MAGNET ASSEMBLY

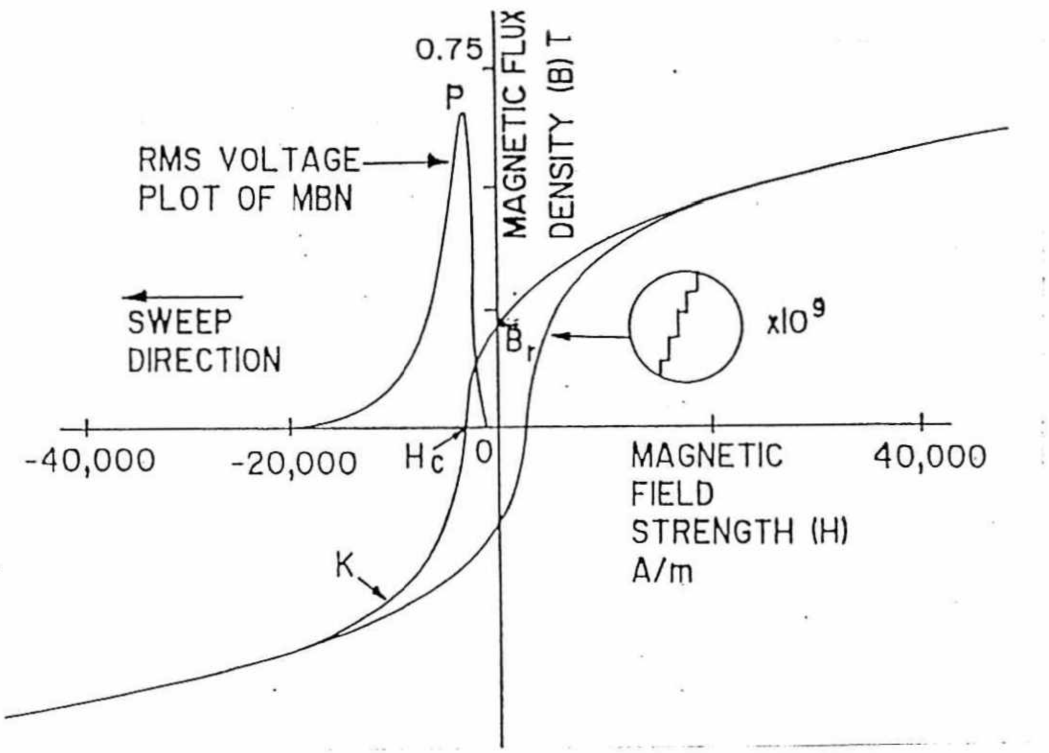


Fig.9: B-H Loop and MBN rms Voltage Plot for a Water Quenched 17-APH Stainless Steel

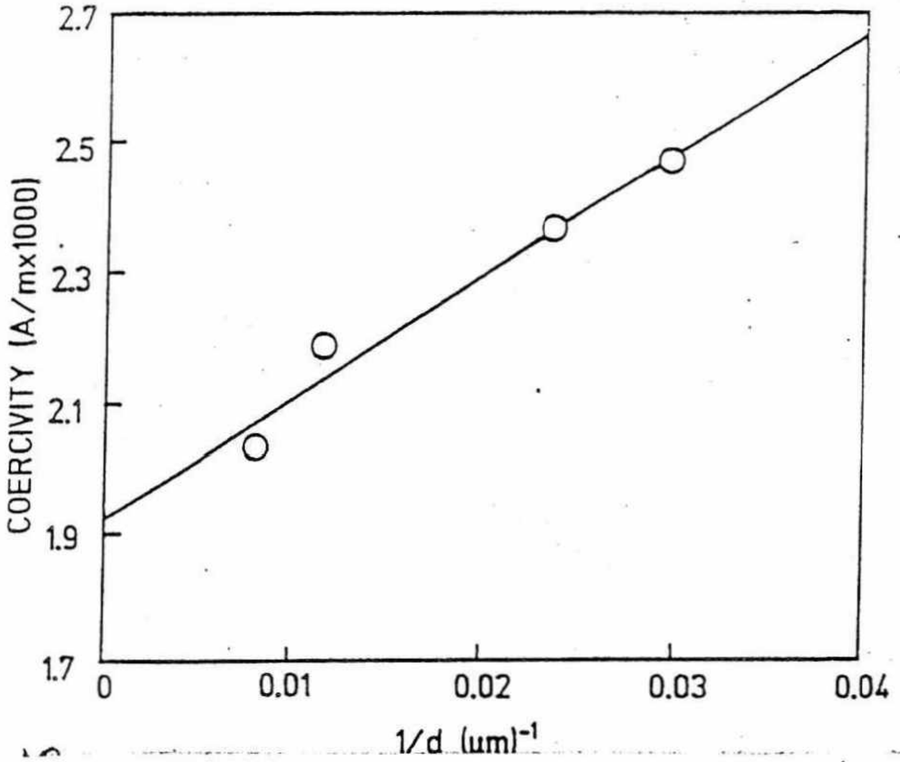


Fig.10: Variation of coercivity in Water Quenched 9Cr-1Mo Steel as an Inverse Function of Prior Austenite Grain Size

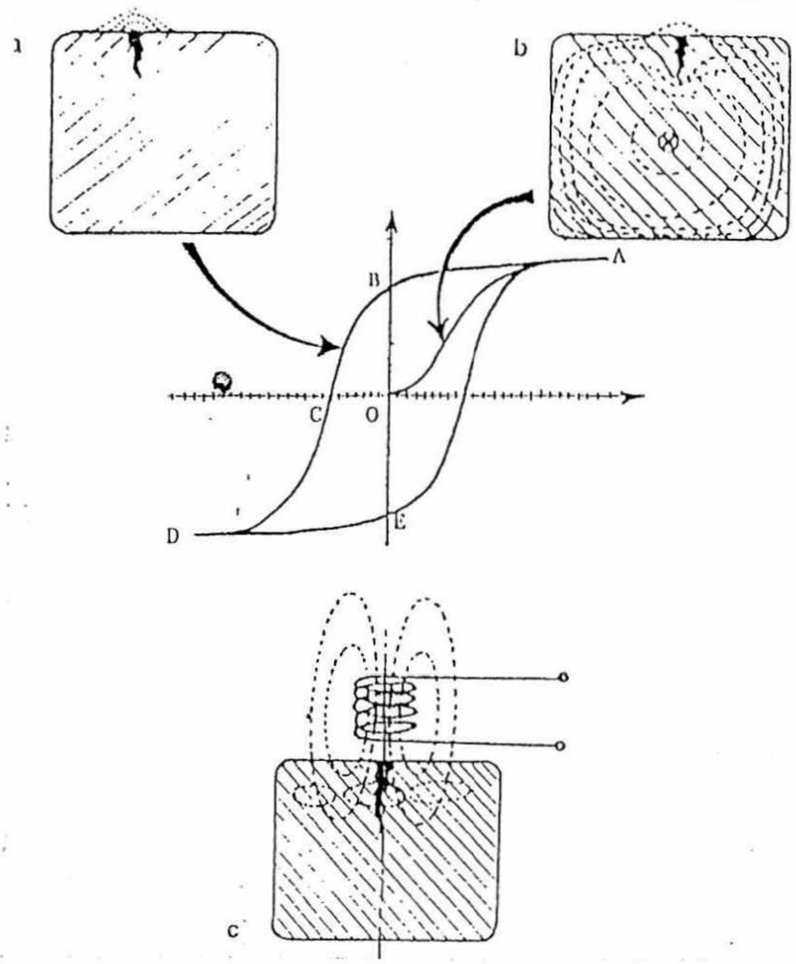


Fig.11: Electromagnetic phenomena utilized in detecting defects in ferromagnetic materials: (a) residual leakage field (region BC); (b) active leakage field (region OA); and (c) ac excitation (region ABCDEA).