

Non-Destructive Techniques for Microstructural and Structural Characterisation

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ABSTRACT

The functional quality of a material or a component is influenced by (i) microstructure (ii) flaws (physical discontinuities like cracks, pores, delaminations *etc.*), and (iii) the presence of stress. Non-destructive evaluation (NDE) techniques are used to control and enhance the quality at various stages of the life cycle of a material or component. In this paper, various NDE techniques are discussed. The techniques mainly discussed are ultrasonics, radiography, X-ray techniques, and acoustic emission. The principles, procedures, advantages, and limitations of each technique, as well as applications in various materials are considered.

INTRODUCTION

Figure 1 showing the various dimensions of microstructural and structural features that may be present in a material or component in various combinations of types, sizes and shapes is well cited [1,2]. Figure. 2 shows the NDT techniques that are possible in various dimension domains [1,2]. Table 1 shows what are the material properties that the various features in Fig. 1 affect and what are the possible NDE techniques that can be used to determine or estimate the material properties [3].

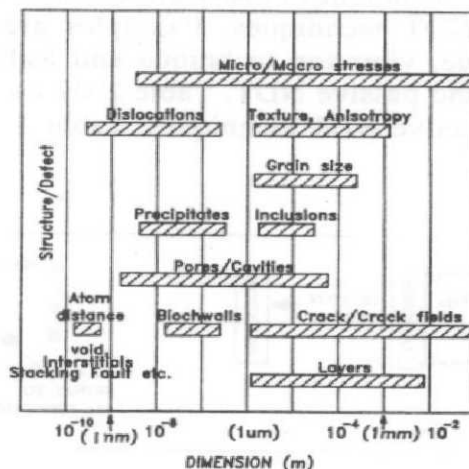


Fig. 1: Dimensions of various microstructural and structural features

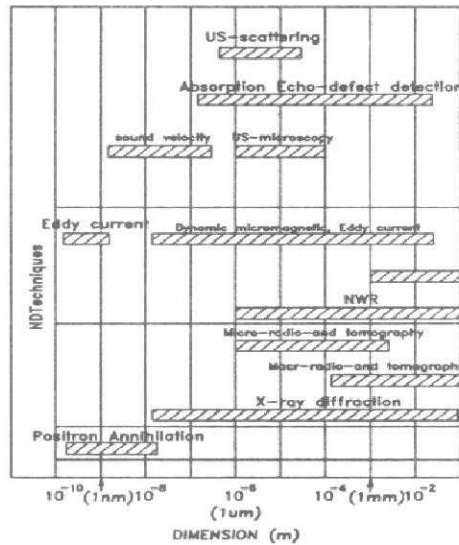


Fig. 2: Various possible non-destructive techniques

ABOUT NDT TECHNIQUES

NDT techniques can be categorised as "active" and "passive" techniques. Figure 3 shows, schematically, principles of these two techniques. Active techniques are those in which a material is interrogated by any of (a) electromagnetic radiation, (b) nuclear radiation, (c) magnetic field, (d) current, or (c) elastic waves. The signal contents of the interrogating medium are changed by way of interaction with the material/component. Suitable detectors are place at suitable locations to detect, process (if needed) and analyse the output signals. In the passive NDT techniques, signals emanate from the components which are detected, processed (if needed) and analysed by suitable detectors. NDT techniques mostly are of the active nature. In comparison, there are fewer passive NDT techniques. Examples are: infra red thermography, acoustic emission technique, vibration technique and leak testing. Fig. 1 shows the principles of active NDT and passive NDT. Table 2 shows various interrogating NDT media and corresponding active NDT techniques. Table 3 shows the basic features of passive NDT techniques.

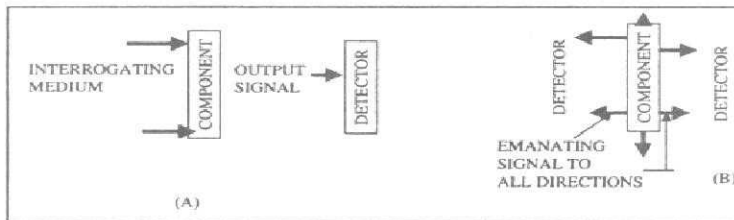


Fig. 3: Schematic sketch showing the principles of (A) active NDT and (B) passive NDT

Table 1: Microstructural and substructural features affecting material properties and the possible NDE techniques to determine and estimate the latter

| <i>Microstructural/ substructural features</i> | <i>Material property affected</i> | <i>Structural, physical characteristics affected</i> | <i>Possible NDE technique</i> |
|---|---|---|---|
| <i>Micro/macro stress</i> | Fatigue, stress corrosion cracking, fracture toughness | Lattice strain Pinning of dislocations Pinning of domain walls Resistivity changes | X-ray diffraction Magnetic Barkausen noise analysis Ultrasonic velocity Laser interferometry |
| <i>Dislocations</i> | All mechanical properties | Resistivity and magnetic property changes Phonon scattering Electron density variations | Eddy current test, magnetic hysteresis loop parameters Ultrasonic absorption Positron annihilation |
| <i>Texture, anisotropy</i> | Preferential mechanical, electrical, chemical and magnetic properties | Preferential crystallographic lattice planes Modulus of elasticity | X-ray diffraction Ultrasonic velocity, ultrasonic attenuation, magnetic Barkhausen noise |
| <i>Grain size</i> | All mechanical properties, corrosion properties, magnetic properties | Ultrasonic scattering Pinning of magnetic Bloch walls by grain boundaries | Ultrasonic attenuation, ultrasonic back scatter Magnetic hysteresis loop parameters Magnetic Barkhausen noise, in-situ microscopy |
| <i>Precipitates/ inclusions</i> | All mechanical properties and corrosion properties | Ultrasonic attenuation mainly by absorption Magnetic properties Modulus of elasticity | Magnetic hysteresis loop parameters Ultrasonic velocity Acoustic microscopy |
| <i>Pore/cavities (present from the fabrication stage)</i> | All mechanical properties, electrical properties, damping characteristics | Modulus of elasticity, density Preferential heat transfer paths Magnetic properties | Ultrasonic velocity Ultrasonic attenuation Acoustic resonance Micro and macro radiography, infrared thermography, magnetic hysteresis loop parameters, MBN, in-situ microscopy |
| <i>Pores/cavities (generated during service)</i> | Manifestations of creep damage | -do- | -do- |
| <i>Atom distance</i> | - | - | Positron annihilation |
| <i>Bloch wall</i> | Magnetic properties | - | - |

Table 1 continued...

| <i>Microstructural/ substructural features</i> | <i>Material property affected</i> | <i>Structural, physical characteristics affected</i> | <i>Possible NDE technique</i> |
|--|---|--|--|
| <i>Crack/crack fields</i> | Component failures through crack growth | Ultrasonic reflection and diffraction, changes in magnetic flux patterns, changes in electrical paths Changes in heat flow patterns, local variation of density | Ultrasonic flaw detection (<i>A</i> -scan, <i>B</i> -scan and <i>C</i> -scan), magnetic flux leakage technique, electric potential drop technique, radiography, acoustic emission, eddy current testing |
| <i>Layers</i> | Through thickness crack growth resistance | Guiding of ultrasonic waves, variation in heat transfer paths, spatial variation of density | Ultrasonic Raleigh wave and other surface specific waves, ultrasonic <i>C</i> -scan, infrared thermography, microfocal radiography |

Table 2: Various interrogating NDT media and the corresponding active NDT techniques

| <i>Interrogating NDT medium</i> | <i>NDT techniques</i> | <i>Type of output</i> |
|---|---|--|
| <i>X-Ray (white radiation)</i> | Microfocal radiography(50-150keV) Conventional radiography (150keV-420keV), High Energy Radiography (in the MeV range), Tomography | Two dimensional shadowgraph Three dimensional shadowgraph (tomography) |
| <i>Gamma rays</i> | Radiography | Two dimensional shadowgraph |
| <i>Neutron, proton</i> | Radiography | Imaging |
| <i>Neutron</i> | Diffraction and stress analysis | Diffraction plot |
| <i>Characteristic X-ray (narrow wavelength range)</i> | Residual stress (RS) measurement. Phase analysis, (<i>e.g.</i> , retained austenite) | $\sin^2 \Psi$ - 2θ plot for RS, magnitude and nature of RS, presence or absence of shear stress. Quantitative data for amount of phase |
| <i>Ultrasonics (elastic wave)</i> | Flaw/defect detection by <i>A</i> -scan (depth location and possible sizing) Flaw/defect detection by <i>B</i> -scan (vertical cross sectional image) Flaw/defect detection by <i>C</i> -scan (horizontal cross sectional imaging) using a synchronous movement of two probes | Oscilloscope display Oscilloscope display Projected view generated by focussed ultrasonic beam in a raster fashion |
| <i>Ultrasonics</i> | SAM (Scanning Acoustic Microscope) | Similar to <i>C</i> -scan imaging but in the reflected mode |

Table 2 continued...

| <i>Interrogating NDT medium</i> | <i>NDT techniques</i> | <i>Type of output</i> |
|---|--|--|
| <i>Ultrasonics aided by laser</i> | SLAM (Scanning Laser Acoustic Microscope) | Imaging of internal features |
| <i>Ultrasonics interrogating and reference beam</i> | Ultrasonic holography | 3 dimensional holographic imaging |
| <i>Current inducing magnetic field</i> | Eddy current testing (with AC) | Change in the impedance of the eddy current probe (X-Y recorder, oscilloscope display or in a two dimensional image, depending on the variations in the presence or absence of flaw and sometimes variations in the material properties) |
| | Magnetic Particle Inspection (with AC or DC) | Pattern developed at surface or subsurface crack regions by magnetic particles (dyed or fluorescent) or by electrical output by a solid state detector of magnetic flux |
| <i>Current</i> | AC or DC potential drop technique for crack depth measurement | Numerical value obtained against calibration |
| <i>Infra red</i> | Active thermography (heating an object) | Infra red image by solid state detectors |
| <i>Microwave</i> | Internal flaw and materials characterisation (in terms of permittivity, dielectric constant) of non-metallic objects | Imaging, metal layer thickness measurement on a non-metallic substrate, bond layer assessment in composites |
| <i>Light</i> | Visual examination (by eye, fibre optics, microscopic lens <i>etc.</i>) | Visual examination (without record), Imaging by film, CCD, TV <i>etc.</i> |
| <i>Laser</i> | Holography, speckle pattern | Imaging |
| <i>UV</i> | Detection of flaw patterns in MPI and penetrant testing | Visual (without record), recording by photography |
| <i>Helium, ammonia gas etc.</i> | Leak testing by active pressurisation | Analog signals by suitable detectors |

ABOUT MATERIALS CHARACTERISATION

The topics "physical and chemical conditions", "microstructure", "mechanical property" and "residual stress" may be considered under the broad area of materials characterisation. In detail, (i) *physical*: dimension, colour, electrical/magnetic/optical properties *etc.* (ii) *chemical*: composition of the material, nature of the material (solid solution, chemical compound *etc.* (iii) *microstructural*: features like, (a) grain size, (b) grain shape, (c) type, size and size distribution of inclusions, (d) relative amounts and

morphologies of various phases, (e) dislocation substructure, (f) crystallographic and grain orientation characteristics (texture) (iv) *mechanical*: (a) properties like yield strength, toughness, fatigue, creep *etc.* Mechanical properties are functions of microstructure and chemical characteristics, (b) Residual stress at the micro (within one or two grains) and macro level (over several grains).

Table 3: Basic features of passive NDT techniques

| <i>Emanating medium</i> | <i>NDT technique</i> | <i>Detection and output</i> |
|---|----------------------|---|
| <i>Acoustic waves due to defect (crack) propagation (100kHz-1MHz)</i> | Acoustic emission. | Piezoelectric crystals, (restricted) capacitive transducer and holographic interferometry |
| <i>Acoustic wave due to leakage of fluid</i> | Leak testing | Ultrasonic/Accelerometer |
| <i>Infra red waves</i> | Thermography | 2 dimensional imaging |
| <i>Displacement (as well as force)</i> | Vibration | Piezoelectric accelerometer. Laser interferometry |

NON-DESTRUCTIVE TECHNIQUES FOR MICROSTRUCTURE

It is well known that microstructure influence material properties-mechanical, chemical and physical. A component performs well, if the designed microstructure is appropriate for the service conditions and no microstructural changes take place in service that are inadequate under the given service conditions.

Traditionally, microstructure has been characterised by optical micrography (OM), scanning electron micrography (SEM), and transmission electron microscopy (TEM). More recently, acoustic microscopy has been introduced for certain applications in which knowledge of the substructures is desired. Specialised microscopy techniques such as scanning tunnelling microscopy, atomic force microscopy *etc.* are not yet relevant for general industrial use.

OM, SEM, and TEM are normally used on small specimens and in the laboratory. TEM often requires very involved procedures for specimen preparation and complex analysis of data. Portable optical microscopes are available which can be used on large components. However, they can give only modest magnifications.

Replica technique has been developed that can be used for obtaining microstructure from actual components by examining the replicas in optical and scanning electron microscope. The replicas of the etched contour of a metallographically prepared surface of an actual component are made into cellulose acetate films. Possibilities of replica technique for carbide extraction from the surface of an actual component has been indicated in the literature but no actual example are available. Since the microstructures are strong functions of thermo mechanical conditions experienced by a material, and since the thermo mechanical conditions may vary from region to region of a component, there is a necessity of the availability of suitable NDT techniques

which can be used to quickly cover the whole surface and volume of a component without being destructive. While this is the situation which is desired, the use of NDT technique(s) is beset with the problem arising from an absence of well understood relationships with the NDT parameters on the one hand, and the microstructural features on the other.

Ultrasonic and magnetic techniques form two of the most important classes of NDT&E techniques, which are used extensively for defect detection/assessment and are presently being developed for materials characterisations for component life assessment. They have high sensitivities and simplicity of operation.

Grain size and mechanical properties in metallic materials – Hall-Petch relationship

The Hall-Petch relation is a semi-empirical relation concerning the yield stress, σ_y , and the grain size, d , of polycrystalline materials. It was discovered by Hall [4] and Petch [5] that the yield stress of steel is enhanced by a decrease in grain size and that this enhancement is characterised by a scaling law,

$$\sigma_y = \sigma_0 + K d^{-1/2} \quad \dots(1)$$

where K is a constant of proportionality, d is the average measured grain diameter and σ_0 is a constant offset of the scaling law often called a friction stress. point (or lower yield stress vs. averaged grain diameter to the $-1/2$ power. Hall demonstrated the relationship for mild steel [4]. Petch demonstrated the relationship for ingot iron, mild steel and spectrographic iron [5]. Since then, the Hall-Petch relation has been very successful and has been found to hold for many different materials. The most common explanation of the Hall-Petch relation comes from the theory of dislocation pileups [6]. Alternate explanation is also available [6]. Apart from yield strength, similar relationships have also been found with impact toughness, tensile strength and hardness. Sometimes, Hall Petch relationship has been found to be valid for dislocation cell size also. No report is available validating the relationship in ceramics and non-metallic composites.

Ultrasonic parameters for grain size and other material parameters

There are two broad classes of ultrasonic parameters, which are useful for NDT for materials characterisation, namely (i) Ultrasonic attenuation and (ii) Ultrasonic velocity [7].

Ultrasonic velocity

The velocity v of an elastic wave like ultrasonic wave can be expressed in the following manner. $v = (M/\rho)^{1/2}$ where ρ is the density and M is an appropriate combination of elastic moduli for the type of wave being propagated. For longitudinal wave in an isotropic body M can be expressed as:

$M_L = E(1 - \epsilon)/\{(1 + \epsilon)(1 - 2\epsilon)\}$, where E is Young's modulus and ϵ is the Poisson's ratio.

For shear waves, we have $M_S = E/\{2(1 + \epsilon)\} = G$, where G is the shear modulus.

It is then clear that whatever microstructural features affect the modulus of a material would affect the ultrasonic velocity. Examples are residual stress, presence of second phase particles particularly cavities and porosities [8].

Ultrasonic attenuation

Elastic waves propagating through an unbounded medium can be expressed as plane waves as follows:

$A = A_0 \exp [i(kx - \omega t)] \exp(-\alpha x)$, where A is the amplitude, A_0 is an initial amplitude at $x = 0$ and $t = 0$, α is the attenuation coefficient, k is the propagation constant which can be expressed as $k = 2\pi/\lambda = 2\pi f/v$ where the term $2\pi f$ is the angular frequency ω . λ is the wavelength, f is the frequency, and v is the velocity.

The attenuation of ultrasonic waves when it passes through a polycrystalline material is mainly due to two effects, namely (i) absorption and (ii) scattering. Both take the energy out of the beam thus resulting in the exponential decay $\exp(-\alpha x)$. The main contribution to scattering comes from the grain boundaries. The following equations are available relating scattering coefficient, grain size and the test frequency.

Raleigh region: For $\lambda \gg 2\pi D$, where D is the average grain diameter, the following equation is applicable

$\alpha = B_1 T \mu^2 f^4$, where B_1 is a coefficient involving the longitudinal and shear wave velocities, μ is the elastic anisotropy of a single grain, T is the appropriate weighted average volume of the grains in the specimen, and f is the test frequency.

Stochastic region: For $\lambda < 2\pi D$ but still not reaching the condition $\lambda \ll D$, the attenuation can be expressed as follows.

$$\alpha = B_2 D \mu^2 f^2 \quad \dots(2)$$

Diffusion region: For $\lambda \ll D$, the attenuation is independent of frequency and can be expressed as

$$\alpha = B_3/D \quad \dots(3)$$

There are several reports on the success of the use of ultrasonic attenuation to estimate the grain size and other mechanical properties.[16-19]

Ultrasonic resonance

Resonance is governed by standing waves where some effective dimension L of the specimen is an integral number of half-wave lengths $L = n\lambda/2$. Since the velocity is related to the frequency and wavelength by $\lambda = v/f$, the resonance frequency can be written as $f_R = nv/2L$

In complicated bodies, the successive resonances are not harmonically related. One can then think of L as being some characteristic length corresponding to that resonance. Since f_R is related to the velocity, it is also related to the moduli. Therefore,

like in the case of velocity the resonance value could be used to characterise a microstructure where porosity and cavities are important features.

Important points in the use of ultrasonics

There are some important points that should be understood before an ultrasonic technique is used for materials characterisation. For attenuation measurement, if a contact probe method is used, then corrections due to the couplant should be known with a desired degree of accuracy if the attenuation coefficient is to be related to the microstructure. One solution is the use of immersion technique in which the component is immersed in a fluid like water and there is a known distance between the probe and the entry point of the ultrasonic wave in the component (Fig. 4). The immersion technique can be used for both flaw detection and materials characterisation. A-scan uses one probe at either ends of the components. On the other hand, C-scan uses two probes at both the ends of the component moving in tandem in a raster fashion

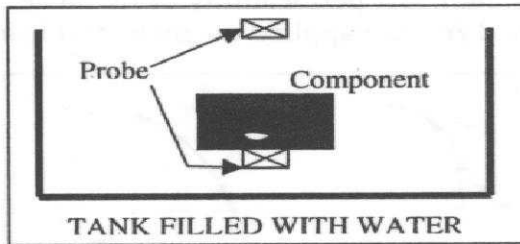


Fig. 4: Sketch showing immersion A-scan and C-scan ultrasonic set up

Another alternative is the use of non-contact methods [8] such as by EMAT (Electromagnetic Acoustic Transducer). EMAT however has a problem that a high frequency more than 5MHz cannot be achieved. For the measurement of grain size, this limitation may perhaps not play a very important role. But for investigation on toughness, this limitation may be important. Another important non-contact mode of generation of ultrasonic waves is by laser excitation [8]. The ultrasonic wave in this case may be detected in a non-contact manner by either an EMAT or by laser interferometry [8].

When ultrasonic velocity is measured, one common difficulty is the knowledge of the thickness of the component through which the ultrasonic wave is propagating. An accepted method to overcome this problem is to use perpendicularly polarised two longitudinal waves and taking the ratio of the velocities. Of course, when surface waves are used, this problem does not exist. Another difficulty in the measurement of ultrasonic velocity is the measurement of ultrasonic transit time of the order of nano seconds. This problem is overcome by the use of digital signal processing by which two chosen ultrasonic back reflection peaks are (in the A-scan mode) are accurately merged by very small shifts in the horizontal time axis and thereby knowing the transit time [9,10]. In the case of ceramics, an added problem comes because of the much higher velocity of ultrasonic waves in ceramic materials. This is overcome by the use of delay blocks. High temperature velocity measurement is often a necessity on

ceramics when such components are in the stage of processing. A quartz acoustic rod between the component and the transducer carries out the measurement [11].

MAGNETIC TECHNIQUES FOR MEASUREMENT OF GRAIN SIZE AND OTHER MECHANICAL PROPERTIES

Magnetic hysteresis loop parameters

Magnetic hysteresis behaviour in a ferromagnetic (and ferrimagnetic) material is a well-known phenomenon, which has been extensively discussed in several treatises [12-14]. Figure 5 shows the schematic representation of the magnetisation processes taking place during a hysteresis sweep. The arrow indicates the direction of magnetisation around the loop. Two basic equations are as follows.

$$B = M + \mu_0 . H \ \& \ B = \mu . H \quad \dots(4)$$

where μ_0 = permeability of vacuum, μ = permeability of the material, B = Induction, M = Intensity of magnetisation, and H = the applied magnetic field strength.

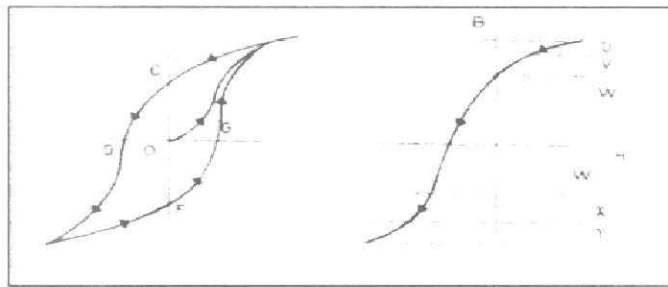


Fig. 5: Schematic sketch showing magnetisation through hysteresis loops

Table 4: Explanations of the terms given in Fig. 5

| Terms | Meaning | Effects and possibilities |
|-------|--|--|
| U | Region of reversible rotation of magnetic moments | - |
| V | Domain nucleation region | - |
| W | Irreversible domain wall displacements mainly of the 180° type | Gives rise to magnetic Barkhausen noise signal. Its peak intensity and temporal intensity variation during magnetisation sweep are important parameters for materials characterisation |
| X | Irreversible domain rotation and annihilation mainly of the 90° type | Gives rise to acoustic Barkhausen noise signal. Has a potential for application in materials characterisation for stress measurement |
| Y | Approach to saturation | - |

The occurrence of the magnetic hysteresis is a consequence of the existence of magnetic domains within a ferromagnetic material. The domains are regions in which the atomic magnetic spins are oriented in the same directions (direction of spontaneous

magnetisation). The domain walls can be classified into 180° walls, in which the spins rotate by 180° from one domain to the other, and 90° walls in which the spin rotates by 90° or so. The various parameters that can be derived from a magnetic hysteresis loop are shown in Fig. 6.

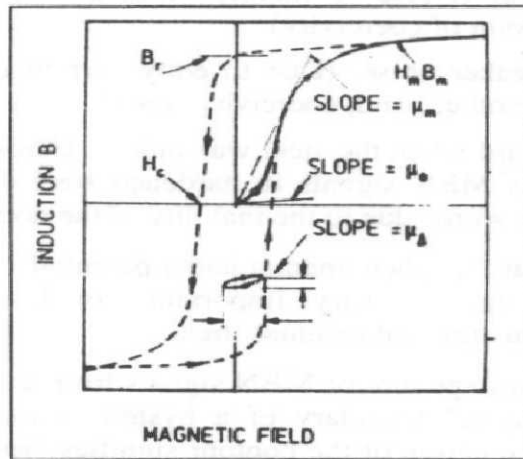


Fig. 6: Parameters that can be derived from hysteresis loop which can be used for characterising microstructural features and mechanical behaviour of materials

The terminology referred to in the above figure are: B_r = retentivity; H_c = coercivity, μ_i = initial permeability; μ_m = Maximum permeability; $\Delta\mu$ = incremental permeability. Besides these, it is possible to use maximum differential permeability, (dB/dH_{max}) which is the maximum slope of $B-H$ hysteresis loop.

Barkhausen noise signal

There are two types of Barkhausen noise signals: (a) magnetic and (b) acoustic.

Magnetic Barkhausen Noise (MBN)

MBN signals are generated when a ferromagnetic or a ferrimagnetic material is magnetised through a hysteresis loop. The source mechanism of this type of signals is mainly the motion of 180° domain walls from one pinning point to another, which changes the local magnetic moment. A sensor coil or an audio head can sense these signals.

MBN discovered by Barkhausen in Germany in 1919 [15] was the first experimental proof of the existence of domain walls. The term "noise" was coined because the existence of signal was discovered in the form of a crackling noise in a telephone speaker. Important observations made by Barkhausen are given below together with comments based on the understanding on MBN signals that we have today. The latter are given within brackets.

- a. The noise was audible. It was audible only after amplification. So arrangements are needed to pre-amplify and amplify the signals before analysing them.
- b. When the magnetic field was varied, the noise was found to be absent in certain range of the variation. (Noise is mainly emitted when the ratio dB/dH is maximum *i.e.*, at the point of coercivity).
- c. Thicker core gave weaker noise. (Due to eddy current damping. So, MBN is surface specific; on the other hand, coercivity is not).
- d. Stronger noise was heard when the steel was milder. Hardened steel did not give any noise at all. (Less MBN signals in hardened steel due to the presence of internal stress. In other words due to the inability of the domain walls to move)

Barkhausen predicted that this phenomenon had a potential for use as an inspection technique. Indeed, it is now a very important non-destructive tool for the characterisation of microstructure and residual stress.

Figure 7 shows an *rms* voltage plot of MBN signals from a 17-4PH steel [16]. The inset shows how the "smooth" boundary of a hysteresis loop would look like if magnified suitably. The step nature of the contour signifies "jerky" motion of domain walls from one pinning point to another. Figure 8 shows a schematic sketch with its essential modules.

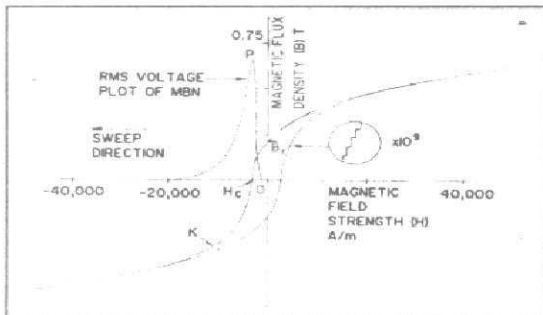


Fig. 7: Plot of *rms* voltage of MBN signals for half of the hysteresis sweep

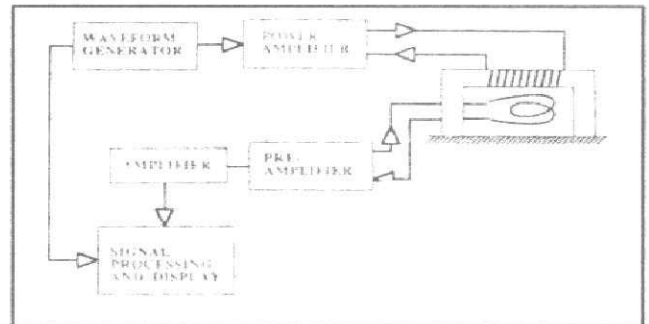


Fig. 8: Block diagram of a typical MBN set up

Acoustic Barkhausen noise

Acoustic Barkhausen noise signals are generated at the knee region of hysteresis loop (point 'K' in Fig. 7). It has not found as much use as MBN, though there are reports of success in using this signal to measure stress as well as grain size.

Coercivity and grain size

Coercivity is normally an inverse function of grain size. Larger grain size means smaller grain boundary areas and less number of domains specially the closure domains of the 90° types which are formed at the grain boundaries and therefore less number of domain walls to move against less number of impeding barriers (including grain boundaries and the domains nucleated from the grain boundaries), and hence

lower coercivity. Opposite arguments can be given for smaller grains. The theories suggest that the impedance to domain wall motion is more important than the difficulty in nucleating reverse domains.

Experimentally, the inverse relationship between coercivity, H_c , and average grain size, d , has been found to be valid for pure iron and low carbon steel [17] and single phase (martensite) alloy steel. [18] *i.e.*, $H_c = k_1 \cdot d^{-1} + k_2$ in which k_1 and k_2 are constants.

For a two phase pearlite-ferrite steel the following equation has been found to be valid [10]:

$$H_c = k_1 \cdot C_f / d_f + k_2 \cdot C_p / d_p \quad \dots(5)$$

where C_f and C_p are the fractions of ferrite and pearlite; d_f and d_p are the average sizes of the ferrite and pearlite grain respectively.

MBN and grain size

Considering that, in many cases, the maximum MBN signal intensity occurs around the point of coercivity, where the ratio dB/dH ratio is maximum, it is reasonable to expect that MBN signal would have a correlation with grain size, because, as has been observed by both theoretical analysis and by experimental observations, coercivity has an inverse relationship with grain size. This relationship comes because the domain wall impediment is more important than domain wall nucleation. On the basis of similar argument, it may be said that MBN signal peak heights should increase with higher grain size, in which case, the grain boundary areas are reduced, and greater mean free paths are available for the domains to propagate once they are nucleated at the grain boundaries.

Coercivity and dislocation density

Various theoretical approaches regarding the nature of interactions of domain walls with dislocation have given a relationship of H with dislocation density of the type: $H_c \propto N^{1/2}$ where N = dislocation density [21].

Coercivity and material hardness

Investigations have been reported on the relationship of magnetic hardness (given by coercivity) and metallurgical hardness. Figure 10 shows the direct and approximately linear relationship between coercivity and hardness of pearlitic steels [20]. Such relationships prompt one to consider coercivity as a monitoring parameter of the metallurgical state of a ferromagnetic steel or an alloy. It is well known that hardness as a parameter is utilised to monitor the microstructural state in many alloy systems - in investigations related to R&D and in industrial practices. Metallurgical hardness is the manifestation of the synergetic effect of a large number of variables such as matrix hardness (solid solution hardening), dislocation density, internal strain, size/shape/volume of secondary phase precipitates, the nature of the interfaces between

the precipitates and matrix, porosities, grain size, *etc.* Most of these microstructural features also influence the coercivity.

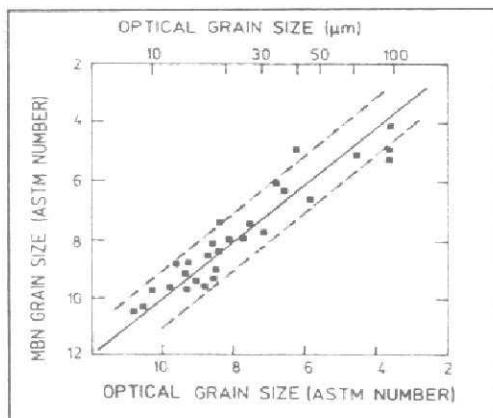


Fig. 9: Linear relationship between grain size from MBN signal and those determined by optical method [19]

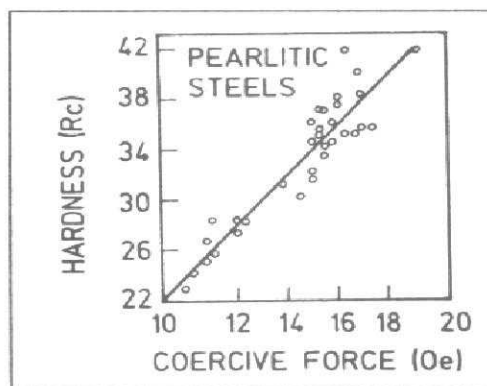


Fig. 10: Linear relationship between magnetic hardness and coercivity) [20]

Magnetic parameters for toughness and creep damage assessment

Though coercivity has been used to assess the hardness of metallic alloys, it has not been reported to have a relationship with toughness (impact or fracture toughness) that could be used in practice. Investigations are needed in this regard.

As regards the assessment of creep damage, it is expected that the magnetic parameters should be well influenced because of the presence of the creep cavities, which like second phase particles should act as pinning points to the movement of the magnetic domain walls. The results so far are however mixed [21]. Definitely, more (and systematic) work is needed in this area.

Apart from the magnetic hysteresis loop parameters and Barkhausen noise signal, there is another kind of magnetic test system, which has been recently introduced. This kind of systems uses magnetic sensors (highly sensitive to detect magnetic leakage flux), like GMR (Giant Magneto Resistance), SQUID (Superconducting Quantum Interference Device) and those based on amorphous magnetic ribbons. These have been found to be sensitive to monitor fatigue damage in certain steels, like in austenitic stainless steel in which ferromagnetic martensitic phase appears as a result of fatigue damage. In the author's laboratory, amorphous magnetic ribbon based sensor has also been found to give excellent sensitivity to monitor the cold work level in austenitic stainless steel in which again, martensitic phase appears as a result of cold work within a matrix of non-ferromagnetic austenite phase [22]. If such systems are successful for ferromagnetic phase in a matrix of non-ferromagnetic matrix, it can be argued that in the reverse case, *i.e.*, in presence of non-ferromagnetic creep cavities in a ferromagnetic matrix, such sensors would be successful to have a relationship with various extents of creep damage. Investigations are needed in this area.

RESIDUAL STRESS MEASUREMENT

Residual stress (also known as locked in stress) is always present in a material because no materials or component can be treated in practice without introducing to some extent or other a differential strain at some location of the materials or the component. The knowledge of residual stress (RS) is important because it affects the fatigue (behaviour under cyclic loading) and stress corrosion cracking properties - the two most important modes of failures of industrial components. Another problem with the presence of residual stress is the distortion that takes place in a component during fabrication and/or service. The knowledge of progressive changes in residual stress is also important to understand the progression of fatigue damage because the changes in the dislocation density and morphology are affected by the progression of the fatigue damage, and the patterns of micro-stress are changed by the former. RS can be categorised as macrostress and microstress. Macrostress is uniform over a large area/volume (say for example several grains). On the other hand, micro-stress is uniform within a smaller area/volume (say within one or two grains).

Residual macro stress measurement by X-ray diffraction [23, 24]

The basic equation in the measurement of RS by X-ray diffraction is the Bragg's law: $n\lambda = 2D \cdot \sin\theta$, where n = the order of diffraction, λ = the wavelength of X-rays used for diffraction, D = the distance of separation between chosen parallel crystallographic planes, and θ = the angle of diffraction.

RS introduces lattice strains, ε . By differentiating the Bragg's law, we get

$$\varepsilon = dD/d_0 = -\cot\theta_0 \cdot d\theta \quad \dots(6)$$

The strain can be measured along a chosen direction by controlling the direction of incidence of the X-rays. For example, if the angle of incidence of the X-rays with respect to the normal to the component surface is ' ψ ', then the strain would be ' ε_{ψ} '. This parameter can be expressed as per the equation given below

$$\varepsilon_{\psi} = [(v + 1)/E] \cdot [\sigma_{\psi} \cdot \sin^2\psi] - (v/E)[\sigma_1 + \sigma_2], \quad \dots(7)$$

where, v = Poisson's ratio, E = elastic modulus, σ_{ψ} = stress along the direction as shown in Fig. 11(a). If an experimental plot is done based on this equation (as shown schematically in Fig. 11(b)), the slope would give the value of ' σ_{ψ} '. The nature of the slope (positive or negative) also would give an idea of the nature of the stress (tensile or compressive).

Residual micro Stress measurement by X-ray diffraction

The FWHM (full width at half maximum) of XRD peaks are strongly affected by the presence of micro-stress, and is an important parameter in this regard [25, 26]

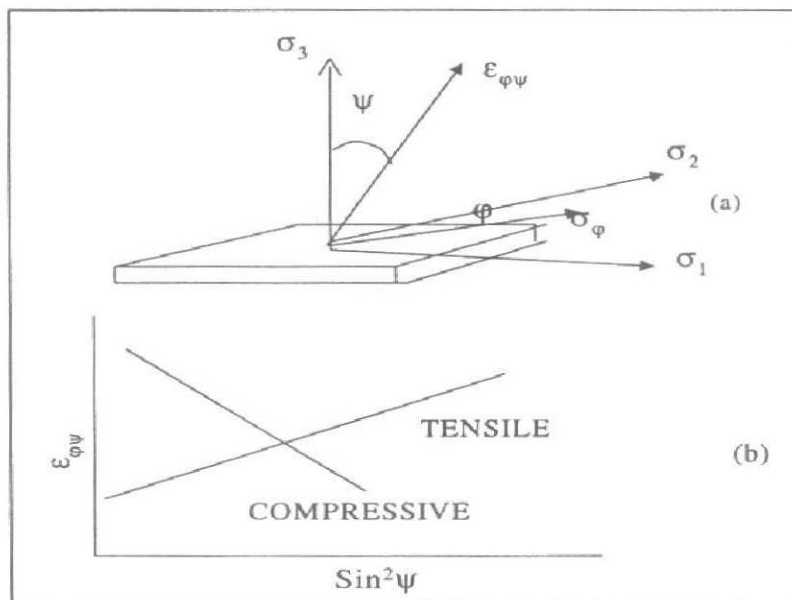


Fig. 11: (a) Geometrical configuration used in the measurement of residual stress by X-ray diffraction technique (b) Typical plot obtained by multi ' ψ ' technique, where ' ψ ' = the angle of incidence of X-rays with the normal to the component surface.

Residual stress measurement by magnetic methods

The discussion earlier in the paper on the relationship between various magnetic parameter and the various microstructural and substructural parameter (like dislocations) is relevant for a discussion on the possibility of the use of magnetic techniques for the measurement of residual stress. The effect of stress on the magnetic hysteresis loop and the difference in the effect from tensile and compressive stress has been known for a long time [13]. Both magnetic and acoustic Barkhausen effect signals have been found often to be suitable for the estimation of residual stress [27]

CONCLUDING REMARKS

An attempt has been made in this paper to discuss some important basic aspects of non-destructive techniques for microstructural and structural characterisation. It has not been possible to discuss in details all the techniques that are possible. Instead, attention has been given to the more important techniques like ultrasonics, magnetics and X-Ray diffraction techniques. However, to the extent possible, mention has been made almost of all the techniques and where they would be important.

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