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Centre for Non-destructive Evaluation at CSIR-National Metallurgical Laboratory, Jamshedpur for materials characterisation and damage evaluation: an Overview

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Abstract:

Non-Destructive Evaluation (NDE) centre established in 2003, with a partial funding from Department of Science & Technology (DST), Govt. of India at CSIR- National Metallurgical Laboratory, Jamshedpur has developed different NDE based methodologies for characterisation of microstructure and assessment of different kinds of damage (creep, fatigue, corrosion etc) for diverse components from aerospace, defence, power and steel sector. Besides, need based development of NDE sensors and techniques for specialised applications are also the integral part of Centre activities. This paper will highlight some of the above activities particularly the research activities on fatigue and corrosion damage evaluation in structural materials by advanced NDE.

Keywords: fatigue, microstructural damage, non-linear ultrasonic, ultrasonic, work rolls

1. Introduction

Research and developments at CSIR-National Metallurgical Laboratory (CSIR-NML) focuses on **Minerals, Metals and Materials**. Through an arsenal of state of the art facilities and infrastructure, and on the strength of its expertise, CSIR-NML has evolved into a premier Indian organization in the stated areas. Its materials evaluation and characterization facilities compare with the best in the world. CSIR-NML has been active for the last two decades on structural health monitoring and remaining life assessment of materials in the power plant, petrochemicals and steel industries both through microstructure and mechanical property evaluation as well as non-destructive evaluation (NDE).

Over the years, NDE activities for component integrity assessment augmented by way of equipment and personnel too. In 2003, with a partial funding from Department of Science & Technology (DST), Govt. of India, a non-destructive evaluation centre for excellence has been established at CSIR-NML. The Centre supported by a wide range of NDE techniques, encompassing ultrasonic, non-linear ultrasonic, acoustic emission, magnetic and electromagnetic and experience in developing NDE protocols for diverse components from aerospace, defence, power and steel sector through focused application oriented research. Need based development of NDE sensors and techniques for specialised applications are also the integral part of Centre activities. The centre over the years has developed different NDE based methodologies for characterisation of microstructure and assessment of different kinds of damage (creep, fatigue, corrosion etc) in various grades of industrial materials [1-3]. CSIR-NML work on creep and ageing behaviour study of Cr-Mo steel using magnetic NDE is well published [4-7].

Another objective of the centre is to provide solutions to the industrial problems. Presently CSIR-NML is also actively involved in providing the solutions to the Work Rolls manufacturers as well as the user industries. This paper will highlight some of the above activities particularly related to the development of methodologies for microstructural and damage evaluation of structural components. Emphasis will be given on fatigue damage evaluation in structural materials using advanced ultrasonic techniques. Paper will also touch upon our activities in solving few industrial problems.

2. Fatigue damage evaluation by non-linear ultrasonic:

In the recent past there is a fast growing interest in the application of nonlinear ultrasonic to NDE for materials characterization and defects detection [8-14]. NLU deals with a broad range of non-linear phenomena of mechanical vibrations and wave propagation in fluids and solids. The nonlinearities in these phenomena may be classified into two categories: classical and non-classical ones. Among the non-classical nonlinearity, it is the nonlinearity resulting from cracks, contacts and interfaces, which show non-symmetric elasticity when ultrasound waves interact with them. The non-symmetric elasticity results in highly nonlinear elastic response to ultrasound waves. In the field of micro-damage diagnostics, this nonlinear ultrasonic has experienced a rapid growth in recent years. Usually material degradation is preceded by some kind of nonlinear mechanical behaviour before significant plastic deformation or material damage occurs. It is known that the microstructural feature which is predominantly altered during fatigue damage progression is a distribution of dislocation which causes significant change in the material's nonlinear elastic wave behaviour. The higher order harmonics generated in the degraded materials, have been attempted to measure by several researchers in various types of materials and has been found that NLU might be a potential non-destructive technique for material degradation evaluation.

CSIR-NML has initiated the research activities on NLU from 2004 onwards and has worked on damage evaluation like fatigue and pitting corrosion in structural materials [1-3,15]. In this paper we will highlight our work on low cycle fatigue damage evaluation in pure iron and carbon steel.

2.1. Experimental

Low cycle fatigue tests on flat dog bone type specimens of pure iron and carbon steel (0.6 wt % C) were performed on an Instron servo-hydraulic testing machine with strain amplitude of ± 0.5%. Test frequency for pure iron was 0.05 Hz whereas for carbon steel it was 0.5 Hz. The average fatigue life of the materials were determined from the LCF tests on five specimens for both the materials and was found 550 cycles for pure iron and 1308 cycles for carbon steel respectively. Interrupted fatigue tests were then performed on pure iron as well as carbon steel specimens with interruptions at 500, 1000, 1200 and 1300 cycles for carbon steel and at 10, 50, 100, 300, 400 and 500 cycles for pure iron. NLU tests were performed on each sample before fatigue testing and after each of the interruption.

For NLU measurement, a high power gated ultrasonic signal analysis device, RAM 5000 (make: Ritec, Warwick) was used. A detail of the RAM 5000 system is described in our earlier paper [15]. 5 MHz longitudinal transducer was used as a transmitter, and a longitudinal broadband transducer (4MHz-16MHz) with a centre frequency at 10 MHz was used as a receiver. The transmitted signal was five cycles tone burst. The received signal from the 10 MHz broadband sensor was filtered using a band pass filter and then amplified by a low-noise preamplifier. The transmitted signal was fed to one of the channels of the NI-PXI digitiser and the amplified received signal was fed to the other channel. Fast Fourier Transformation (FFT) was performed on the 1st reflected echo of the received signal for all the measurements.

The nonlinear ultrasonic parameter (β) was determined after measuring the amplitude of the fundamental (A_1) and 2nd harmonic (A_2) from the FFT using the relation,

$$\beta = \frac{8v_L^2 A_2}{\omega_0^2 z A_1^2} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where, z is the sample thickness, v_L is the longitudinal velocity and ω_0 is the excitation signal frequency. In general, for same sample, different amplitude pairs (A_1 and A_2) were obtained by using stepped attenuators to vary the input power and the slope of the line A_2 vs A_1^2 was used in the equation to find β. This method can only be applied at the laboratory on small specimens but can not be practically feasible for online monitoring of NLU parameter during component testing. So we have developed the online NLU parameter determination system keeping the transmitting power constant throuout the measurement on each specimen. Figure 1 shows the experimental setup for the NLU measurement.

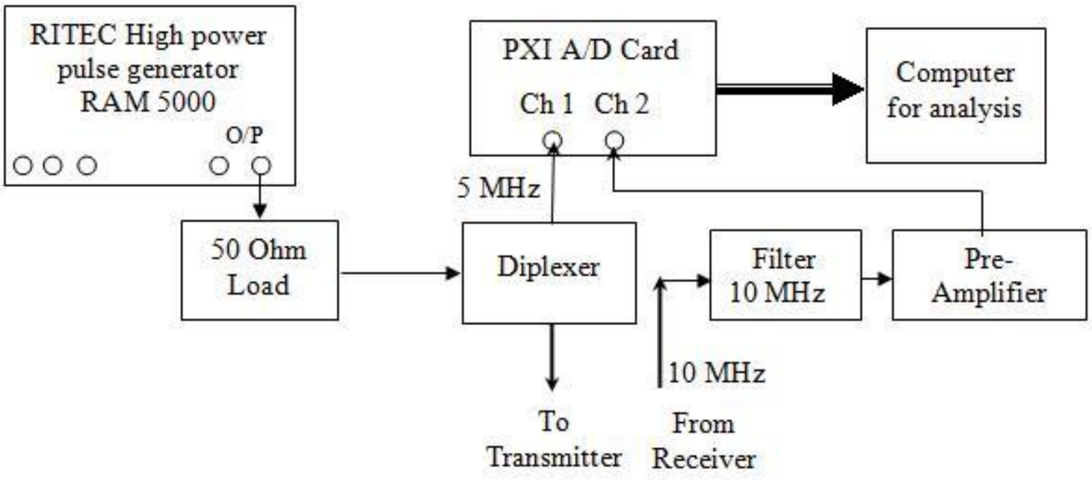


Figure 1: Experimental setup for non-linear ultrasonic parameter measurement

For microstructural characterization, TEM investigation was performed on the damaged specimens using Philips CM200 at 200 KV. The TEM specimens were prepared from the transverse section of the samples cut from the gage portion. A slow speed diamond cutting tool and mechanical polishing were used to thin the specimens to 0.1mm. Final thinning was and electro polishing was made using a twinjet electro-polishing unit employing a solution of glacial acetic acid and Perchloric acid at a ratio of 9:1. Microstructures at various stages of interrupted fatigue cycles were recorded to observe the dislocation morphology.

2.2. Results & Discussions:

NLU parameter was measured prior to fatiguing and after each interrupted fatigue cycles at the central location of the gage length region. Since the nonlinearity produced due to the change in dislocation morphology due to fatigue is much smaller than that produced from instrumentation and from the PZT transducers, we have determined normalized NLU parameter $\Delta\beta/\beta_0$ at each fatigue cycle, where, β_o is the value of NLU parameter before fatiguing and Δβ is the difference in β before and after fatiguing. By normalizing the NLU parameter with respect to the initial value, we could nullify the contribution of nonlinearity by the factors other than that due to fatiguing. The variation of $\Delta\beta/\beta_0$ with number of fatigue cycles with the corresponding microstructures has been plotted in figure 2.

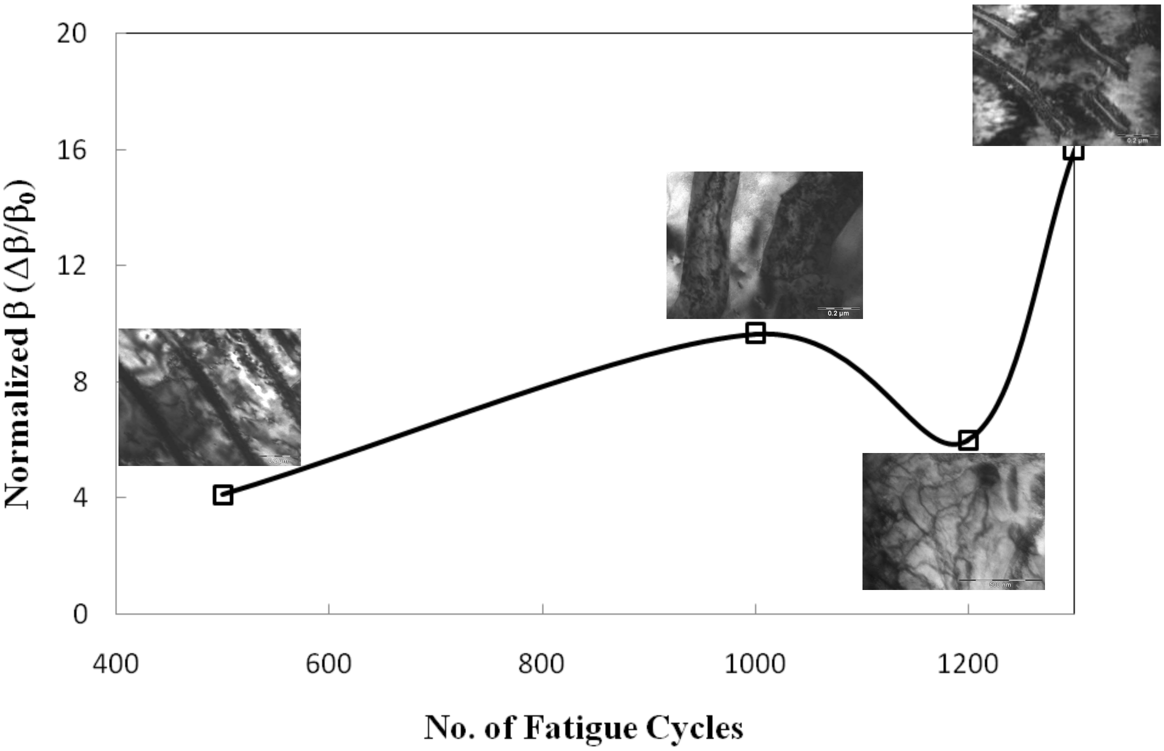
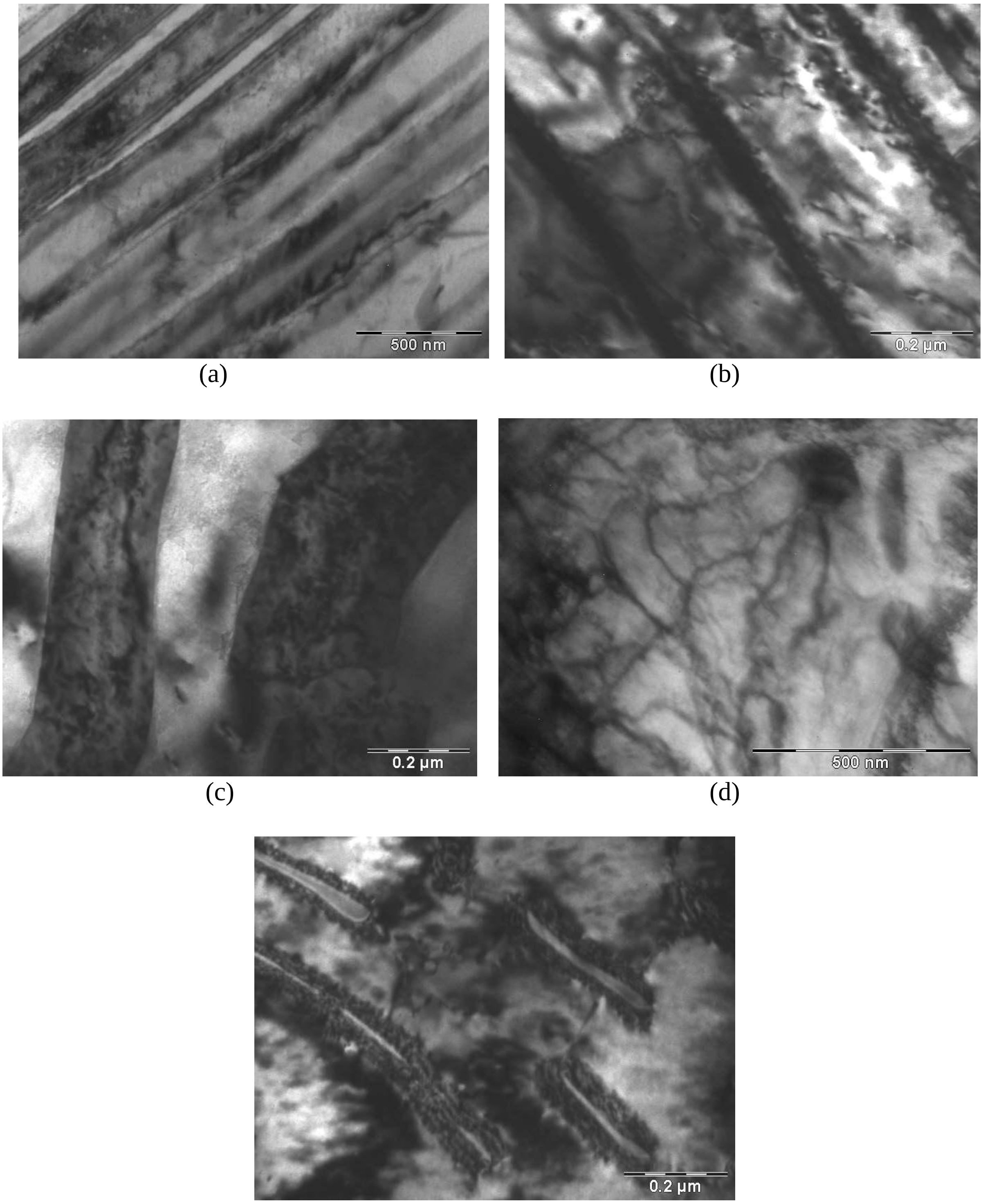


Figure 2: Variation of normalised β with fatigue cycles

It is observed from figure 2 that the quantity, $\Delta\beta/\beta_0$, increases up to 1000 cycles, after that it drops and again starts increasing from 1200 cycles. There is a sharp increase in $\Delta\beta/\beta_0$ at 1300 cycles. To analyze the variation in $\Delta\beta/\beta_0$ at different fatigue cycles, TEM investigations have been carried out to reveal the microstructures at various stages of interrupted fatigue cycles. TEM micrographs of as-received and after 500, 1000, 1200 and 1300 fatigue cycles are shown in figures 3(a)-(e) respectively. At the annealed condition, the microstructrure consisted of alternative bands of pearlite and cementite as depicted in figure 3(a), typical of high carbon steels as in this investigation. After imposition of 500 fatigue cycles, high dislocations were observed in the ferrite matrix (figure 3(b). Increase in dislocation density was observed with further fatiguing for 1000 cycles (figure 3(c)), after 1200 cycles, incomplete dislocation cell formation was noticed within the ferrite matrix (figure 3(d)). When the fatigue cycle is increased to 1300, breaking of cementite laths with associated dislocation clouds were observed as shown in figure 3(e).



(a) (b) (c) (d) (e)

Figure 3: TEM micrographs of fatigued specimens (a) annealed, (b) 500 cycles, (c) 1000 cycles, (d) 1200 cycles and (e) 1300 cycles

Ferrite-pearlitic lamellar structure is typical of high carbon steels and the volume fraction of each of these constituent phases is determined by the carbon content and the processing conditions employed during manufacture of the steel. Accordingly, the dislocation generation and cell formation in pearltic steels is liable to be controlled by the relative fractions of the soft ferrite and hard cementite phases and their inter-lamellar spacing, among other parameters. Accordingly, a more prominent dislocation cell structure could be observed in the pro-eutectoid ferrite matrix of this steel. As with increased fatigue cycle (say at 500 and1000 cycles), the dislocation density is increased, the quantity $\Delta\beta/\beta_0$ also increased. Further increase in fatigue cycles (after 1200 cycles) resulted in a refinement of dislocation substructure. The dislocation veins in the interior of the sub-structures disappeared resulting in a decrease in the quantity $\Delta\beta/\beta_0$. Application of further fatigue cycles (1300) resulted in the deformation of cementite in pearlite through shearing by nearly 45^o to the loading axis by bending or kinking (fig 3e). This event is reflected by the sharp increase in $\Delta\beta/\beta_0$.

Besides correlating the NLU parameter with microstructural features, which is a destructive and time consuming technique, an approach has been attempted to quantify the damage with respect to total energy stored after each interruption. Since the low cycle fatigue has been carried out at constant strain amplitude, peak stress after each interruption was correlated with the normalized NLU parameter for both carbon steel and pure iron and is shown in figure 4 and 5 respectively.

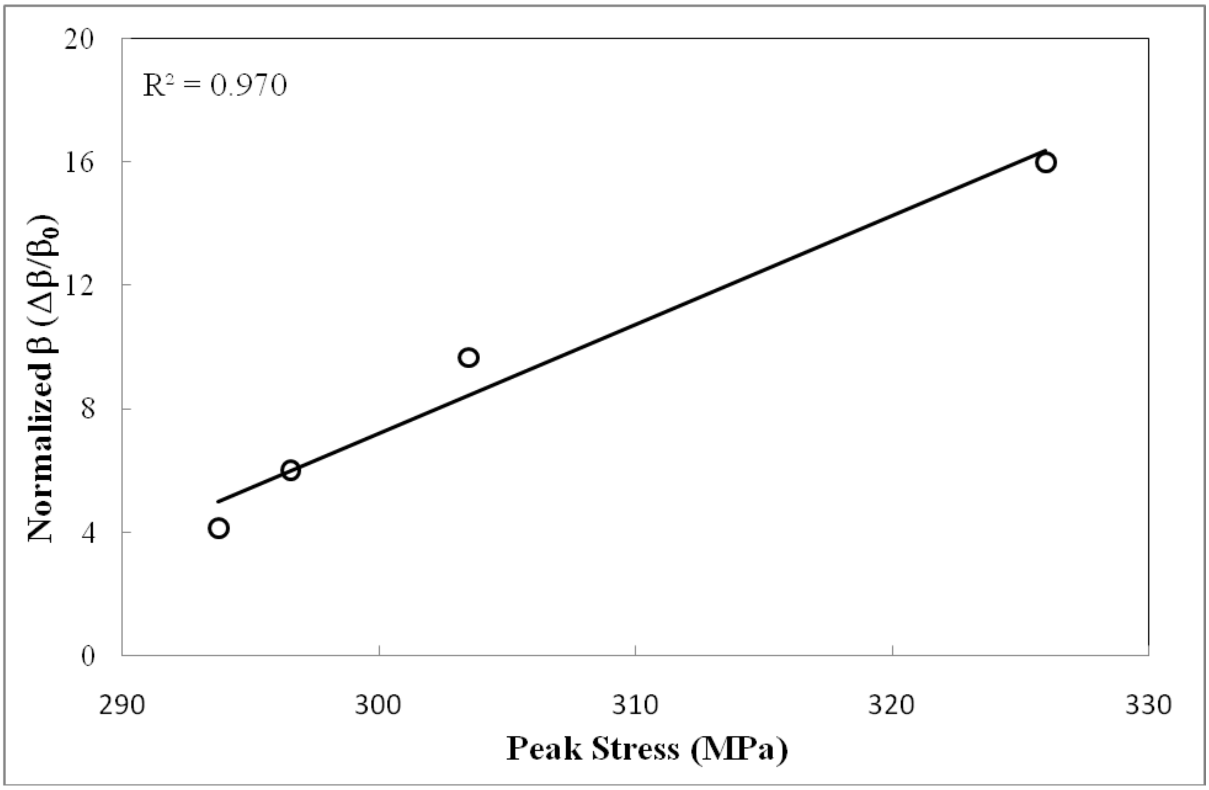


Figure 4: Variation of normalized β with peak stress in carbon steel with 0.6wt% C

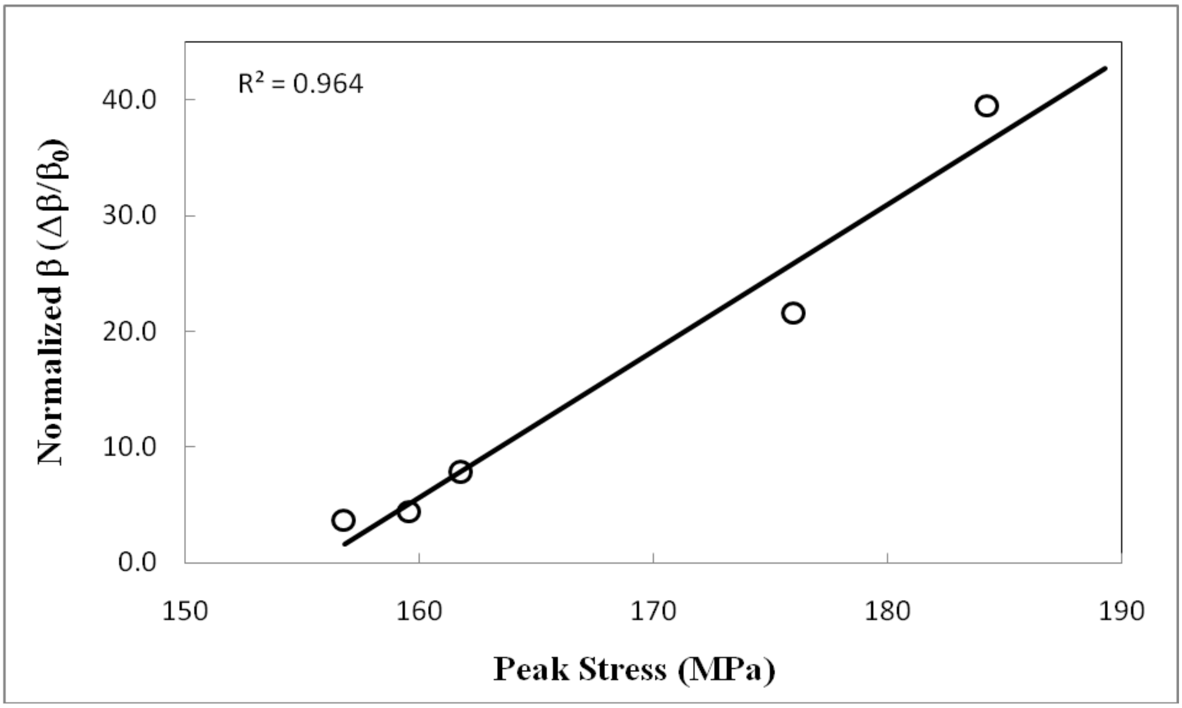


Figure 5: Variation of normalized β with peak stress in pure iron

Normalized β shows a good linear correlation with peak stress for both pure iron and carbon steel with 0.6 wt% C. Work has also been extended for carbon steel with 0.3 wt% carbon and a similar linear relationship with normalized β and peak stress was observed. Rate of increase in β with damage is higher for pure iron compared to carbon steel.

3. Health Monitoring of Work Rolls:

The quality of work rolls plays a very important role for the roll mills as well as to the flat products. This is because of the considerable economic losses incurred during emergency shutdowns of a roll mill due to break up of rolls [16-18]. The use of non-destructive testing during both manufactures and exploitation of rolls as well as during scheduled reconditioning of rolls is the realistic way to ensure the maximum efficiency of a mill and to increase the quality of the products. At CSIR-NML we have developed the methodology to ensure the bond quality (with or without flaws) of Indefinitely Chilled Double Poured (ICDP) rolls manufactured by vertical centrifugal casting process using ultrasonic technique. This work will be useful for any ICDP roll manufacturer for the quality assessment of shell-core bonding of rolls without any false alarm and will reduce the rejection of rolls. Besides, a surface wave based ultrasonic technique has been developed to detect fine cracks on the barrel surface of High Speed Steel (HSS) rolls as well as to find the optimal grinding condition for a complete crack free roll surface for the roll mills. A brief of the methodologies is described in this article.

3.1. Methodology development for quality assessment of shell-core bonding of ICDP rolls without any false alarm

"Indefinite Chill Double Poured (ICDP)" work rolls are produced by centrifugal casting and have outer shell made of indefinite chill white cast iron and core is of spheroid graphite cast iron. During casting of rolls, the aim is to achieve full metallurgical bond between the shell and the core. Any disbonding, presence of flux/ slag at the interface or excess of carbides, microporosities, *graphite flakes*, non-metallic inclusions may reduce the strength of the bond and that ultimately leads to roll failure. Hence it is a general practice of Roll manufacturer to assess the interface quality of rolls using non-destructive technique especially ultrasonic. The shell/core interface echo in ultrasonic testing is an indicator for measuring bond quality. A good bond roll will show a lower interface echo, whereas a higher interface echo is observed for poor bond. But at the interface, there is always a possibility of non-uniform mixing which may lead to non-uniform distribution of graphite in the form of nodules as well as flakes. Because of non-uniform distribution of graphite in the form of flakes, ultrasonic wave velocity and attenuation will vary. Hence while checking by ultrasonic technique for the soundness of the shell-core bond, the interface echo (IF) amplitude may mislead the interpretation of result and many times IF amplitude crosses the acceptable amplitude % for flawless bond. This in turn causes rejection of the rolls.

We have taken 3 pieces cut from a new roll for our experiment and one test sample is shown in figure 6 below:

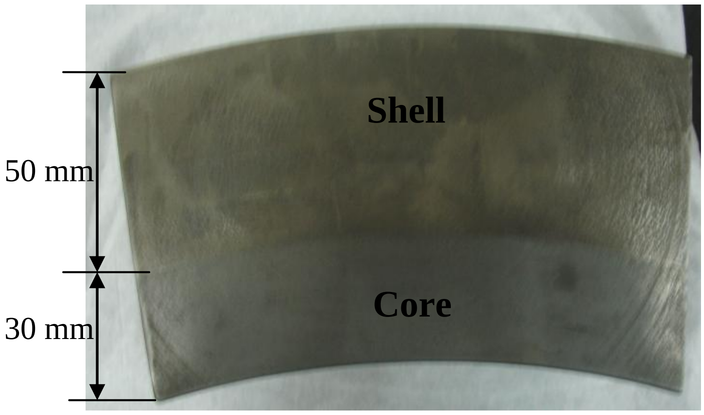
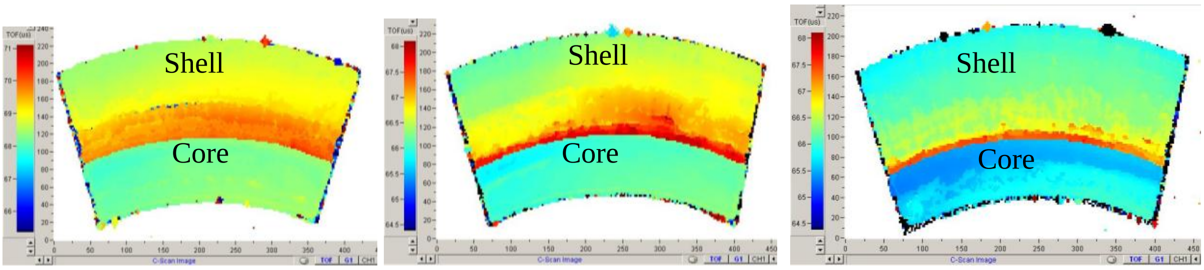


Figure 6: A white cast (shell) and SG cast iron (core) centrifugally bonded sample

Ultrasonic immersion technique for C scan imaging was carried out on the test sample using a 5 MHz focused immersion transducer with a spatial resolution of 200 μm. The focal point of the probe was adjusted on the surface of the specimen. The C scan was carried out to determine the time-of-flight/velocity of ultrasonic wave at the shell, core and the interface zones. A non-uniform velocity distribution with varying width has been observed at the near interface region in all three samples though the samples were taken from the same region of the roll. Figure 7 shows the ultrasonic c-scan images of 3 samples in the TOF mode. It has been observed that the velocity in the near interface zone is varying significantly and non-uniformly in all three samples.



Sample #1 Sample #2 Sample #3

Figure 6: Ultrasonic c-scan images in TOF mode

In sample #1, near the interface a uniform 20 mm wide, in sample #2 a non-uniformly varying and in sample #3, a narrow low velocity zone was observed near the interface The velocity of ultrasonic wave in the near interface zone was determined from the A scan signal of the some location. A-scan signal at one such location of sample #2 is shown in figure7.

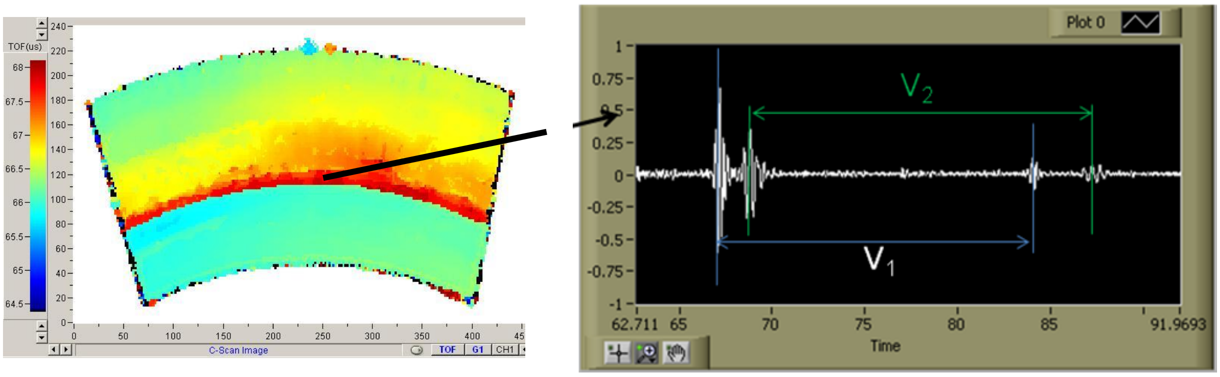


Figure 7: A scan signal as received from a particular location of sample #2

It was found that in the interface zone ultrasonic signal corresponds to two different velocities; V_1= 5564 m/s and V_2= 5264 m/s. While measuring the interface echo amplitude from the top of the sample to assess the quality of bonding, this non-uniform low velocity near-interface zone plays a vital role. In general, the ultrasonic longitudinal velocity of the white iron region was 5490 m/s and that of SG cast iron region was 5600 m/s. Due to the presence of low velocity region (ranging from 5031 m/s to 5300 m/s) near the interface, the reflected energy of ultrasonic wave from the interface varies.

Ultrasonic Pulse/Echo technique was used to measure the interface echo (IF) amplitude of the samples. The 2 MHz longitudinal probe was placed at the top surface of each sample and the IF echo amplitude was recorded for different positions with a constant gain. Result of IF echo amplitude as measured for sample #2 is shown in figure 8.

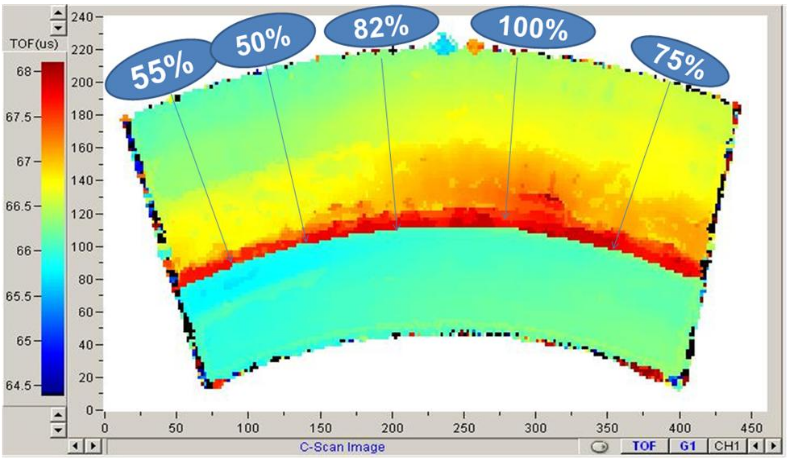


Figure 8: IF echo amplitude as measured from the top of the samples at different positions

Variation in IF echo amplitude was observed from position to position. The amplitude of interface echo was correlated with the observed low velocity near interface regions. It was observed that the IF amplitude is high (100%) in the location where low velocity near interface zone was quite wide even though there is no flaw whereas it was low (50%) corresponds to the location with narrow low velocity zone.

4. Concluding Remarks

There have been consistent attempts by CSIR-NML to develop methodologies for microstructural and damage assessment of structural components by various non-destructive evaluation techniques. The CSIR-NML over the years has developed different NDE based methodologies on magnetic, ultrasonic and non-linear ultrasonic for characterisation of microstructure and assessment of different kinds of damage such as creep, fatigue, corrosion etc. This paper highlights only fatigue damage evaluation in pure iron and carbon steels using non-linear ultrasonic. Presently strong emphasis has also been given to develop improved NDE techniques for solving various industrial problems one such is presented in this paper.

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References

1. S Palit Sagar, S. Das, N. Parida and D.K. Bhattacharya, D K, ‘Non-linear ultrasonic technique to assess fatigue damage in structural steel’, Scripta Materialia, Vol. 55, No. 2. pp. 199-202, 2006

2. Santanu De, S Palit Sagar, Swapna Dey, Amit Prakash and I. Chattoraj, ‘Quantification of pitting in two tempers of 7075 aluminium alloy by non-destructive evaluation’, Corrosion Science, Vol. 52,No. 5. pp. 1818-1823, 2010.

3. S Palit Sagar, A.K. Metya, M. Ghosh and S. Siva Prasad, ‘Effect of microstructure on non-linear behavior of ultrasound during low cycle fatigue of pearlitic steels’, Materials Science and Engineering A, Vol. 528,No. 6. pp. 2895-2898, 2011.

4. J.N. Mohapatra, J. Swaminathan, M. Ghosh and A. Mitra, ‘Magnetic Nondestructive Evaluation of Creep Behavior in Water-Quenched Modified 9Cr-1Mo Steel’, Met. and Mater. Transactions A, Vol. 41, No. 4. pp. 900-905, 2010.

5. J.N. Mohapatra, N. R. Bandopadhyay, M.K. Gunjan and A. Mitra, ‘Study of high-temperature ageing and creep on bainitic 5Cr–0.5Mo steel by magnetic NDE techniques’, Journal of Magnetism and Magnetic Materials, Vol. 322, No. 6. pp. 589-595, 2010.

6. J. N. Mohapatra, A. K. Panda, and A. Mitra, ‘Magnetic properties evaluation of ageing behaviour in water-quenched 5Cr-0.5Mo steel’, Journal of Physics D-Applied Physics , Vol. 42, No. 9. 095006, 2009.

7. A. Mitra, J. N. Mohapatra, J. Swaminathan, M. Ghosh,A.K. Panda and R. N. Ghosh, ‘Magnetic evaluation of creep in modified 9Cr–1Mo steel’, Scripta Materialia, Vol. 57, No. 9. pp. 813-816, 2007.

8. H. Cantrell and W. T. Yost, Acoustic harmonic generation from fatigue induced dislocation dipoles. Philos Mag A 1994;69(2):315–26

9. A. Hikata, C. Elbaum, ‘Generation of ultrasonic second and third harmonics due to dislocations’, Phys Rev., Vol 144, pp 469, 1966.

10. K.Y. Jhang, K. C. Kim, ‘Evaluation of material degradation using nonlinear acoustic effect’, Ultrasonics Vol.37, pp 39–44,1999.

11. P.B. Nagy, ‘Fatigue damage assessment by nonlinear ultrasonic materials characterization’, Ultrasonics, Vol. 36, pp. 375–381, 1998.

12. W. T. Yost, Jr. J .H Cantrell, M.A. Breazeale, ‘Ultrasonic nonlinearity parameters and third order elastic constants of copper between 300 and 3K’, J Appl Phys. Vol. 52, No. 1, pp. 126–128, 1981.

13. J.Y. Kim, L.J Jacobs, J. Qu, ‘Experimental characterization of fatigue damage in a nickel base super-alloy using nonlinear ultrasonic waves’, J Acoust Soc Am., Vol 120, No. 3, pp. 1266–1273, 2006.

14. W.L. Morris, O. Buck, R. V. Inman, ‘Acoustic harmonic generation due to fatigue damage in high strength aluminium’, J Appl Phys, Vol. 50, No. 11, pp. 6737–6741, 1979.

15. A.K. Metya, N. Parida, D. K. Bhattacharya, N.R. Bandopadhyay and S. Palit Sagar, ‘Assessment of Localized Plastic Deformation during Fatigue in Polycrystalline Copper by Nonlinear Ultrasonic’, Metal.nd Materials Transactions A, 38 (12). pp. 3087-3092.

16. M. Hashimoto, T. Oda, K. Hokimoto, ‘Development and application of high speed tool steel rolls in hot strip rolling’, Nippon Steel Technical Report, pp. 66:82, 1995.

17. P. Hetchman, J. Ballani, R. Zorn, 36th Mechanical Working and Steel Processing, Iron & Steel Society, Vol. 32, pp 21, 1994.

18. H. Takada, K. Yamamoto, H.Hiraoka, ‘Quality measurement techniques for hot rolling rolls’, JFE Technical Report, Vol. 10,pp 42, 2007.