CHARACTERIZATION OF MECHANICAL BEHAVIOR OF Cr-Mo STEELS USING MINIATURE SPECIMEN TESTING APPROACH

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by

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CERTIFICATE

This is to certify that the thesis entitled "**Characterization of Mechanical Behavior of Cr-Mo Steels Using Miniature Specimen Testing Approach"** being submitted by **Mr.Kannan D.S. Chandrasekaran** to the **Indian Institute of Technology Delhi, (India)** for the award of the degree of Doctor of Philosophy in Applied Mechanics Department is a bonafide research work carried out by him under our supervision and guidance.

The research reports and the results presented in this thesis have not been submitted in parts or in full to any other University or Institute for the award of any degree or diploma.

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Kannan Chandrasekaran

ABSTRACT

Integrity evaluation and Life assessment of in-service components call for the knowledge of current mechanical and microstructural health requiring large amount of sampling of the material and retrofitting. Retrofitting through welding and post weld heat treatment of pressure vessels especially with large wall thicknesses can induce unwanted defects / damages in addition to being cumbersome and imposing extended non-availability of the facility. In addition, life assessment based on either virgin properties or indirectly estimated from in-situ metallography / hardness measurements result in erroneous predictions or strongly conservative values leading to premature retirement / replacements. Therefore, there is an immense interest in the process industries in general and refining industry in particular for accurate evaluation of current mechanical properties of in-service components of low alloy steels through miniature specimen test techniques for condition monitoring of critical facilities during shut downs.

Several researchers have proposed a variety of miniature specimen test techniques applied to the various industrial components and materials using different sample geometries and thicknesses. Among these, Small Punch Test (SPT) technique has evolved as a predominant technique with the potential of providing several mechanical properties such as strength, ductility, ductile – brittle transition temperature (DBTT) and fracture toughness with very little volume of material. The technique still remains non-standard with scope for development of empirical correlations specific to the class of steels, sample thickness range and configuration of test fixtures. In addition, it is seen that small punch test technique has not been explored for mechanical

characterization of embrittlement of Cr-Mo steels in presence of entrapped hydrogen. Hence, a need has been felt to develop test techniques and correlations for assessment of mechanical properties and toughness of Cr-Mo steels with hydrogen embrittlement through small punch test technique.

In the present study, metallurgical characterization and mechanical property evaluation of four different grades of Cr-Mo steels namely 1Cr0.5Mo (Service exposed), 1.25Cr0.5Mo (Service exposed) 1.25Cr0.5Mo (Virgin) and 2.25Cr1Mo (Virgin) has been carried out using standard samples. The chemical composition, microstructural analysis, tensile tests, charpy impact tests, micro hardness, 'J' integral fracture toughness test, J-R curve and tearing modulus against ductile crack growth have been evaluated for these materials.

A SPT fixture has been designed, fabricated and used for the present investigation to test circular disc shaped specimens of 0.3mm, 0.4mm and 0.5mm thicknesses using hemispherical tipped punches of 1.2mm, 1.7mm, 2.2mm and 2.5mm diameters. The setup has been used on an Instron universal testing machine with a laser sensor for accurate measurement of minute displacement of the specimen. An EDM wire cut process has been used for the fabrication of miniature specimens followed by surface preparation prior to testing. Small punch tests on the chosen materials for all combinations of punch diameter and sample thicknesses have been carried out.

The load – displacement response has been categorized into five distinct regions corresponding to the elastic bending (P_y,d_1, E_1), plastic bending (P_2, d_2, E_2), membrane stretching (P_3,d_3,E_3), instability (P_m,d_4,E_4) and fracture (P_5, d_5, E_5) of the sample where

a) P_y,P_2, P_3,P_m, P_5 , b) d_1,d_2, d_3,d_m,d_5 and c) E_1,E_2,E_3,E_4,E_5 represent the load, displacement and energy values at yield, transition of elastic bending – plastic bending, transition of plastic bending membrane stretching, maxima and point of fracture respectively. These parameters as well as the displacements and energies corresponding to membrane stretching (d_{23}, E_{23}), instability (E_{34}, d_{34}) and fracture (d_{45}, E_{45}) regions have been identified using an innovative approach for data analysis.

The individual parameters have been studied with respect to punch diameter, sample thickness as well as standard mechanical properties. The most significant parameters reflecting the mechanical properties have been identified. Sectional metallographic studies on the fractured small punch test samples and interrupted tests have been conducted to understand the geometrical effects of SPT as well as micro mechanism of deformation. The effects of punch diameter, sample thickness and material properties on various SPT parameters in relation to the deformation mechanisms have also been explained.

The typical variations of (i) yield load P_y and E_1/d_1 with square of thickness, (ii) peak load P_m and E_{34}/d_{34} with product of punch diameter and thickness, (iii) the product of (P_m+P_5) with d_{45} with the square of thickness and (iv) the energy of fracture region E_{45} with square of thickness have been studied. Based on this study, load based and energy based correlations for estimation of yield strength, tensile strength as well as 'J' initiation fracture toughness of Cr-Mo steels has been proposed. The proposed correlations in the present investigation have been compared with the reported correlations in literature. The correlation proposed in the present work are found to be in good agreement with the standard experimental results and resulted in minimum error.

Based on the studies, optimum SPT procedure and parameters for life assessment have been suggested.

A procedure for thermal charging of hydrogen into standard and miniature samples using a tubular reactor of a high pressure hydrocracker pilot plant and its characterization has been established and standardized. Hydrogen charging into the miniature and standard samples of 2.25Cr1Mo steel up to four different levels has been carried out after electroless nickel plating process. Samples have been characterized for bulk hydrogen concentrations by melt extraction method. The standard and small punch tests on the hydrogen charged samples have been carried out and the mechanical and fracture properties have been obtained. The significant SPT parameters have been compared for various levels of charged hydrogen. In addition, fractographic studies on the tensile and fracture specimens have also been carried.

At higher hydrogen levels of around 15ppm, a significant decrease in tensile strength, yield strength, initiation fracture toughness $J_{0.1}$ and tearing modulus for crack initiation are seen when tested at a crosshead speed of 0.2mm/min. The individual trends of the SPT parameters with respect to specimen thickness and punch diameters have been studied and compared with that of uncharged condition. Hydrogen charging did not seem to change the micro mechanism of deformation in SPT. The applicability of earlier proposed correlations for estimation of tensile strength and yield strength of Cr-Mo steels for hydrogen charged conditions has been verified. A specific correlation for estimation of initiation fracture toughness $J_{0.1}$ has been proposed for hydrogen charged 2.25Cr1Mo steels. The important findings of the present investigation have been summarized at the end.

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