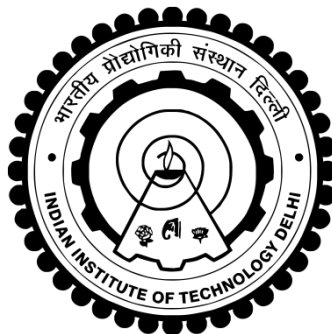


**EVALUATION OF MECHANICAL PROPERTIES OF HEATER TUBE
MATERIALS OF REFINERIES AT VARIOUS TEMPERATURES USING
MINIATURE SAMPLE TESTING APPROACH**

SATYA PAL SINGH



**DEPARTMENT OF APPLIED MECHANICS
INDIAN INSTITUTE OF TECHNOLOGY DELHI
FEBRUARY, 2015**

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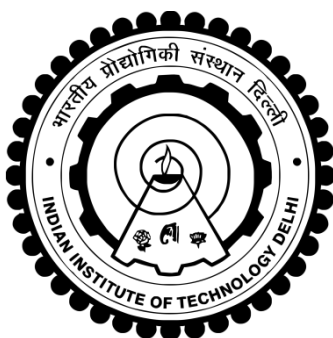
by

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Submitted in fulfilment of the requirements of the degree of
Doctor of Philosophy

to the



INDIAN INSTITUTE OF TECHNOLOGY DELHI

FEBRUARY, 2015

CERTIFICATE

This is to certify that the thesis entitled “Evaluation of Mechanical Properties of Heater Tube Materials of Refineries at Various Temperatures Using Miniature Sample Testing Approach” being submitted by Mr Satya Pal Singh to the Indian Institute of Technology Delhi, India for the award of the degree of Doctor of Philosophy in the Department of Applied Mechanics is a bonafide research work carried out by him under our supervision and guidance.

The research reports and the result 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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(Satya Pal Singh)

ABSTRACT

The mechanical properties (such as YS , UTS, DBTT, creep , toughness etc) of service-exposed components alter with the cumulative time & temperature of exposure and environment and therefore safe and economic operation of the process plants needs the quantitative assessment of mechanical properties at regular intervals. The conventional methods of determination of mechanical properties of metals require bulk metal removal leading to process shutdown and post repair. There has been constant emphasis for many years now on quantitative assessment of current mechanical properties of the components for reliable health assessment and Remaining Life assessment (RLA). To achieve such an objective, small punch test (SPT) / Miniature sample test technology (MSTT) has emerged as one of the promising techniques.

SPT requires a very small quantity of metal which can be removed almost non-destructively from component surface to form a miniature sample and performing a destructive test on the miniature sample using specialized miniature sample testing facility to assess mechanical properties of parent metal. Such techniques being almost nondestructive in nature requires no post sampling treatment, leading to no process time loss. However, significant part of such studies has revolved around irradiated materials of nuclear industry and those used in refining and other process industries have been studied only to a limited extent. Moreover such studies have been revolving around the estimation of room temperature (RT) properties of materials with the help of empirical correlations. Estimation of high temperature mechanical properties has been attempted but no empirical relations are available in literature.

Heater tube is one such critical application in petroleum and many other process industries wherein the tubes of various grades of CrMo steels are operated even beyond 700 °C skin temperature and are subjected to high temperature damages like creep, carburization, microstructural degradation etc and hence calls for their periodic health assessment. Under the present work, two most widely used grades of heater tube steel namely 5Cr0.5Mo and 9Cr1Mo having service life of nearly 23 years and 10 years respectively have been selected for study at room temperature and elevated temperature ranging from 400 °C to 700 °C through small punch test.

Initially the selected materials have been thoroughly analyzed and evaluated for their mechanical properties including yield strength (YS), ultimate tensile strength (UTS) and ductile brittle transition temperature (DBTT) using standard test methods. These parameters are set as bench mark parameter for their comparison with SPT parameters.

A small punch test set up has been in-house designed and developed. Innovatively designed SPT set up consist of less than 1 cm³ of inert gas charged space to avoid oxidation of sample and punch during its prolonged use and can be suitable for small punch creep tests also.

Subsequently, small punch test method, suitable for elevated temperature, has been developed and tests carried on two service exposed heater tube grade materials namely 5Cr0.5Mo and 9Cr1Mo. The load displacement data recorded during SPT tests has been analyzed and relationship developed between SPT parameters and standard mechanical properties for estimation of YS and UTS. Two different approaches, namely energy and load based have been adopted for such relation development and both of the two found equally capable. In the present

research, both the materials have been found to correlate well with common relationships for each of the property (i.e. YS and UTS) in the entire range of test temperature.

Significant light has been thrown on the parameters affecting the results of small punch test, particularly carried out at elevated temperatures. Many aspects related to deformation mechanism of SPT have also been discussed. Inter relationships among the various SPT parameters have also been explored.

Low temperature studies have been carried out using liquid nitrogen cooled environmental chamber and an in-house developed testing set up. The standard DBTT obtained with Charpy V Notch tests have been compared with the results of small punch low temperature and relationship developed for estimation of DBTT of heater tube grade steels by SPT. Both the materials have been found to correlate well with a common relationship.

TABLE OF CONTENTS

CAPTION	PAGE NO.
CERTIFICATE	i
ACKNOWLEDGEMENTS	ii
ABSTRACT	iv
TABLE OF CONTENTS	vii
LIST OF FIGURES	xiv
LIST OF TABLES	xxxii
LIST OF ABBREVIATIONS	xxxix
 CHAPTER -1: INTRODUCTION	 1-10
1.1 Preface	1
1.2 An overview to a petroleum refinery	2
1.3 Refining equipments and material of construction	3
1.4 Process heaters	3
1.5 Material damage mechanisms	6
1.6 Necessity of health / remaining life assessment	7
1.7 Role of miniature specimen technologies for health / life assessment of components	9
 CHAPTER -2: LITERATURE REVIEW	 11-48
2.1 Introduction	11
2.2 An overview of CrMo steels	11
2.3 Problems of Cr-Mo steels in high temperature environment	12
2.3.1 Creep	12

CAPTION	PAGE NO.
2.3.2 Micro structural aging	13
2.3.3 Carburization	14
2.3.4 Oxidation	15
2.3.5 Corrosion	15
2.3.6 Hydrogen attack	16
2.4 Health / remaining life assessment methodology in oil refineries	16
2.5 Miniature specimen testing approach	17
2.5.1 Miniaturised tensile test	19
2.5.2 Miniaturized Impact Test	20
2.5.3 Punch Test Techniques	22
2.5.4 Estimation of mechanical properties using small punch test	27
2.5.5 Estimation of fracture properties using small punch test	31
2.5.6 Application of small punch test in high temperature tensile properties	37
2.5.7 Application of small punch test in creep	38
2.5.8 Application of small punch test in non metallic materials	39
2.5.9 Small punch test modelling and simulation studies	40
2.6 Some limitations of the literature available correlations	41
2.7 Need of standardisation of small punch test method	42
2.8 Advantages of small punch test technique	44
2.9 Inferences from the literature review	45
2.10 Motivation for the present Study	46
2.11 Objectives of the present Study	47

CAPTION	PAGE NO.
CHAPTER -3: MATERIALS, MICROSTRUCTURE & MECHANICAL PROPERTIES	49-87
3.1 Introduction	49
3.2 Materials chosen for study	49
3.2.1 5Cr0.5Mo service exposed (ASTM A213 grade T5)	51
3.2.2 9Cr1Mo service exposed (ASTM A213 Grade T9)	52
3.3 Laboratory assessment of the materials	55
3.3.1 Visual observations and thickness measurement	55
3.3.2 Chemical composition analysis	57
3.3.3 Hardness of materials	58
3.3.4 Microstructural characterization of materials	60
3.3.4.1 Microstructure of 5Cr0.5Mo(SE)	60
3.3.4.2 Microstructure of 9Cr1Mo(SE)	61
3.3.5 Determination of standard tensile properties	65
3.3.5.1 Specimen preparation	65
3.3.5.2 Test equipment and procedure of testing	65
3.3.5.3 Result interpretation	68
3.3.6 Charpy impact test	70
3.3.7 Micro-fractographic studies	71
3.3.7.1 General procedure	71
3.3.7.2 Fractographic studies on tensile samples of 5Cr0.5Mo(SE)	72
3.3.7.3 Fractographic studies on tensile samples of 9Cr1Mo(SE)	78

CAPTION	PAGE NO.
3.3.7.4 Fractographic studies on impact samples	85
CHAPTER – 4: DESIGN & DEVELOPMENT OF SMALL PUNCH TEST (SPT) SETUP	88-102
4.1 Introduction	88
4.2 Experimental Setup for Small Punch Tests	88
4.2.1 SPT Setup and Accessories	88
4.2.2 Material Selection for fabrication of SPT and Heat treatment	91
4.2.3 Design of dies holder	92
4.2.4 Design of lower and upper dies	93
4.2.5 Design of punch	93
4.3 Testing Machine and Accessories	100
4.4 Fabrication and preparation of small punch test specimens	102
CHAPTER -5: SMALL PUNCH TEST METHODOLOGY AND EXPERIMENTAL RESULTS	103-145
5.1 Introduction	103
5.2 Selection of test materials test temperatures	104
5.3 Test Procedure	104
5.4 Small Punch Test: General Observations	107
5.5 Determination of load and displacement parameters of SPT	115
5.6 Determination of energy parameters	119
5.7 Estimation of mechanical properties from SPT	120
5.7.1 Load based estimation of Yield strength	122
5.7.2 Energy based estimation of Yield strength	125

CAPTION	PAGE NO.
5.7.3 Load based estimation of ultimate tensile strength (UTS)	127
5.7.4 Energy based estimation of ultimate tensile strength (UTS)	130
5.8 Comparison of proposed correlations with literature existing correlations	134
5.9 Study of scatter in the estimated YS and UTS in present study	137
5.10 Verification of correlations proposed for estimation of mechanical properties	138
5.11 Verification of correlations proposed for estimation of mechanical properties at RT with higher sample thickness	143
CHAPTER -6: RESULTS AND DISCUSSIONS ON MINIATURE	146-214
SPECIMEN TESTS	
6.1 Introduction	146
6.2 Studies on SPT Parameters	146
6.2.1 Elastic bending regime	149
6.2.2 Plastic bending regime	154
6.2.3 Membrane stretching regime	166
6.2.4 Instability regime	177
6.2.5 Fracture regime	187
6.2.6 Investigation into the behavioural pattern of SPT with temperature	193
6.2.7 Broad conclusions from studies on SPT parameters	197
6.3 Sectional studies on SPT sample	199

CAPTION	PAGE NO.
6.4 Microstructural studies on SPT samples in fracture zone	205
6.5 Fractographic studies	207
6.5.1 Fractographic studies on 5Cr0.5Mo(SE)	207
6.5.2 Fractographic studies on 9Cr1Mo(SE)	208
CHAPTER -7: LOW TEMPERATURE STUDIES	215-245
7.1 Introduction	215
7.2 Charpy impact test system	215
7.3 Test method	216
7.4 Determination of standard DBTT	218
7.4.1 Studies on 5Cr0.5Mo(SE) material	221
7.4.2 Studies on 9Cr1Mo(SE) material	227
7.5 Fractographic studies on Charpy V Notch test samples	232
7.6 Cryogenic studies on SPT and determination of DBTT	236
7.7 Interrelationship between standard DBTT and cryogenic temperature SPT	243
CHAPTER -8: CONCLUSIONS AND RECOMMENDATIONS	246-260
8.1 Summary of the present work	246
8.1.1 Inferences from the characterization of Cr-Mo grade heater tube steels considered for study	246
8.1.2 Inferences from design and development of small punch test (SPT) set up	248
8.1.3 Inferences from small punch test methodology and experimental results	249
8.1.4 Inferences from damage behaviour of SPT	253

CAPTION	PAGE NO.
8.1.5 Inferences from the low temperature studies	256
8.2 Philosophical conclusions	259
8.3 Recommendations for further studies	260
REFERENCES	261-274
APPENDIX 'A'	275-280
APPENDIX 'B'	281-296
BRIEF BIODATA OF THE AUTHOR	297