

学校编码：10384

分类号_____密级_____

学号：32420110153847

UDC _____

厦 门 大 学

博 士 学 位 论 文

反应堆用马氏体时效强化钢的微观组织与性能研究

A Microstructure and Mechanical Property Study of
Maraging Steel PH13-8Mo in Nuclear Applications

黄子敬

指导教师姓名： 李宁 教授

专业名称： 核科学与工程

论文提交日期： 2015 年 4 月

论文答辩日期： 2015 年 5 月

学位授予日期： 2015 年 12 月

答辩委员会主席： _____

评 阅 人： _____

2015 年 5 月

厦门大学博硕士学位论文摘要库

**A Microstructure and Mechanical Property Study of
Maraging Steel PH13-8Mo in Nuclear Applications**

A Dissertation Submitted for the Degree of Doctor of Philosophy

By

Zijing Huang

Supervised by

Prof. Ning Li

College of Energy

Xiamen University

厦门大学博硕士学位论文摘要库

厦门大学学位论文原创性声明

本人呈交的学位论文是本人在导师指导下,独立完成的研究成果。本人在论文写作中参考其他个人或集体已经发表的研究成果,均在文中以适当方式明确标明,并符合法律规范和《厦门大学研究生学术活动规范(试行)》。

另外,该学位论文为(李宁教授)课题(组)的研究成果,获得(李宁)课题(组)经费或实验室的资助,在(加州大学伯克利分校)实验室完成。(请在以上括号内填写课题或课题组负责人或实验室名称,未有此项声明内容的,可以不作特别声明。)

声明人(签名):

年 月 日

厦门大学博硕士学位论文摘要库

厦门大学学位论文著作权使用声明

本人同意厦门大学根据《中华人民共和国学位条例暂行实施办法》等规定保留和使用此学位论文，并向主管部门或其指定机构送交学位论文（包括纸质版和电子版），允许学位论文进入厦门大学图书馆及其数据库被查阅、借阅。本人同意厦门大学将学位论文加入全国博士、硕士学位论文共建单位数据库进行检索，将学位论文的标题和摘要汇编出版，采用影印、缩印或者其它方式合理复制学位论文。

本学位论文属于：

（ ） 1. 经厦门大学保密委员会审查核定的保密学位论文，
于 年 月 日解密，解密后适用上述授权。

（ ） 2. 不保密，适用上述授权。

（请在以上相应括号内打“√”或填上相应内容。保密学位论文应是已经厦门大学保密委员会审定过的学位论文，未经厦门大学保密委员会审定的学位论文均为公开学位论文。此声明栏不填写的，默认为公开学位论文，均适用上述授权。）

声明人（签名）：

厦门大学博硕士学位论文摘要库

目录

中文摘要.....	i
英文摘要.....	iii
第 1 章 前言	1
1.1 核能发展概况.....	1
1.2 反应堆用结构材料概况.....	1
1.3 马氏体时效钢.....	4
1.4 核材料开发中的新技术.....	7
1.5 选题意义与技术框架.....	10
第 2 章 实验材料及实验方法	13
2.1 试验材料及热处理工艺.....	13
2.2 金相试验.....	13
2.3 X 射线衍射检测.....	14
2.4 维氏硬度试验.....	14
2.5 纳米压痕试验.....	14
2.6 聚焦离子束加工技术.....	15
2.7 三维原子探针样品分析.....	20
2.8 拉伸试验.....	21
2.9 辐照试验.....	21
第 3 章 马氏体时效钢的室温组织分析	22
3.1 时效试样的金相分析.....	22
3.2 时效试样的 X 射线衍射结果与分析.....	26
3.3 时效试样的相变分析.....	28
3.4 三维原子探针样品检测结果.....	29
3.5 本章小结.....	33
第 4 章 马氏体时效钢的室温性能检测与分析	35
4.1 试验准备.....	35
4.2 时效试样的维氏硬度.....	39
4.3 参考样品的纳米压痕检测.....	40
4.4 样品辐照损伤后的纳米压痕测试结果.....	43

4.5	本章小结	46
第5章	马氏体时效钢组织内的析出物研究	47
5.1	三维原子探针样品制取	47
5.2	数据处理与分析方法	48
5.3	时效样品中析出物的三维原子探针分析	50
5.4	600°C 时效 2000h 样品的扫描透射电子显微镜分析	56
5.5	时效样品中的析出物强韧化机制	57
5.6	本章小结	62
第6章	马氏体时效钢高温力学性能检测与分析	64
6.1	实验准备	64
6.2	马氏体时效钢的高温硬度测试	67
6.3	马氏体时效钢的高温蠕变测试	68
6.4	马氏体时效钢的高温拉伸测试	71
6.5	本章小结	73
第7章	马氏体时效强化钢与抗辐照合金钢的对比	75
7.1	抗辐照合金钢发展概况	75
7.2	马氏体时效强化钢与抗辐照合金钢的析出物对比	77
7.3	马氏体时效钢与抗辐照合金钢的高温拉伸性能对比	78
7.4	小结	80
第8章	结论与展望	81
8.1	论文结论	81
8.2	本文创新	82
8.3	未来展望	82
	参考文献	84
	攻读学位期间取得的研究成果	90
	致谢	91

Table of Contents

CHINESE ABSTRACT	i
ENGLISH ABSTRACT	iii
CHAPTER 1 INTRODUCTION	1
1.1 DEVELOPMENT OF NUCLEAR ENERGY	1
1.2 STRUCTURAL MATERIALS FOR NUCLEAR APPLICATIONS.....	1
1.3 MAR-AGING STEELS	4
1.4 NEW TECHNIQUES IN THE R&D OF NUCLEAR MATERIALS	7
1.5 OBJECTIVES AND TECHNICAL FRAMEWORK	10
CHAPTER 2 EXPERIMENTAL MATERIALS AND METHODS	13
2.1 MATERIALS AND HEAT TREATMENT PROCESS.....	13
2.2 METALLOGRAPHIC TEST.....	13
2.3 X-RAY DIFFRACTION	14
2.4 VICKERS HARDNESS	14
2.5 NANOINDENTATION	14
2.6 FOCUS ION BEAM TECHNIQUE	15
2.7 3 DIMENSION ATOM PROBE.....	20
2.8 TENSILE TEST	21
2.9 IRRADIATION TEST	21
CHAPTER 3 MICROSTRUCTURE ANALYSIS OF MAR-AGING STEELS	22
3.1 METALLURGICAL ANALYSIS	22
3.2 X-RAY DIFFRACTION ANALYSIS	26
3.3 PHASE TRANSFORMATION ANALYSIS	28
3.4 THREE DIMENSION ATOM PROBE ANALYSIS	29
3.5 SUMMARY	33
CHAPTER 4 MICROSTRUCTURAL AND MICROMECHANICAL AT ROOM TEMPERATURE.....	35
4.1 EXPERIMENTAL PREPARATION	35
4.2 VICKERS HARDNESS	39
4.3 NANOINDENTATION ON REFERENCE SAMPLES	40
4.4 NANOINDENTATION ON IRRADIATED SAMPLES	43
4.5 SUMMARY	46

CHAPTER 5 ANALYSIS ON PRECIPITATES IN MAR-AGING STEELS.....	47
5.1 PREPARATION OF 3DAP SAMPLES.....	47
5.2 DATA PROCESSING METHODS.....	48
5.3 SCANNING TRANSMISSION ELECTRON MICROSCOPY ANALYSIS.....	50
5.4 STRENGTHENING MECHANISM OF PRECIPITATES	56
5.5 STRENGTHENING MECHANISM OF PRECIPITATES	57
5.6 SUMMARY	62
CHAPTER 6 MECHANICAL TESTING AND ANALYSIS OF MAR-AGING	
STEELS AT ELEVATED TEMPERATURE	64
6.1 EXPERIMENTAL PREPARATION	64
6.2 NANOINDENTATION TEST AT ELEVATED TEMPERATURE	67
6.3 NANOINDENTATION CREEP TEST AT ELEVATED TEMPERATURE.....	68
6.4 TENSILE TEST AT ELEVATED TEMPERATURE	71
6.5 SUMMARY	73
CHAPTER 7 COMPARISON BETWEEN MAR-AGING STEELS AND THE	
IRRADIATION RESISTANCE MATERIALS	75
7.1 DEVELOPMENT OF IRRADIATION RESISTANCE MATERIALS	75
7.2 CHARACTERISTICS OF PRECIPITATES.....	77
7.3 TENSILE STRENGTH.....	78
7.4 SUMMARY	80
CHAPTER 8 CONCLUSION AND THE FUTURE EXPECTATION	81
8.1 CONCLUSION OF THE THESIS.....	81
8.2 INNOVATIVE OF THE THESIS.....	82
8.3 EXPECTATION AND FUTURE WORK	82
REFERENCES	84
PUBLICATIONS	90
ACKNOWLEDGEMENT	91

摘要

马氏体时效强化不锈钢具有杰出的机械性能，且在高温服役环境中表现稳定，因而在航空和传统能源领域广受青睐。此外，材料的热膨胀系数均匀，良好的抗腐蚀性能以及时效前优良的加工特性也引起了核能业界的关注。由于时效后的材料内部能够析出大量细小弥散的 NiAl 金属间化合物，形成吸收辐照损伤空位的势阱，为材料的耐辐照性能创造了客观条件，马氏体时效强化不锈钢也成为了重要的新一代反应堆备选结构材料。

本论文从研究马氏体时效强化不锈钢 PH13-8Mo 的长周期高温性能变化出发，在 450、500 和 600°C 分别对材料进行了长达 2000h 的时效处理，通过扫描电镜技术 (SEM) 和 X 射线衍射技术 (XRD) 等，对不同时效状态下的样品进行了组织表征与相分析，发现材料在 450°C 达到硬度峰值后不发生性能衰减现象，而 500 和 600°C 时效样品中析出物的粗化以及反相奥氏体的出现是材料失效的重要原因。

采用直线加速器设备，使用能量为 1.5MeV 的质子在 300°C 下对固溶态样品和 500、600°C 时效样品进行辐照，累积辐照损伤约为 5dpa。采用纳米压痕设备对样品进行显微力学测试，发现材料在 450°C 时效状态下尺寸效应最为突出。辐照样品的纳米压痕结果显示，固溶态和 600°C 时效样品在 300°C 辐照环境中发生热力学析出，辐照效应加深了热力学析出的过程。500°C 时效样品的辐照硬化现象最不明显，材料具有最优抗辐照性能。

采用三维原子探针检测技术重构了 500 和 600°C 辐照样品辐照区与未辐照区的三维图像，对析出物的成分分析和表征结果表明，离子辐照会促进 600°C 时效试样中 NiAl 析出物的形成与长大，辐照区中析出物的尺寸和密度均大于未辐照区域。另一方面，对变形机制的研究表明，500°C 时效样品的析出物强化通过 Orowan 机制实现，600°C 时效样品的强化则通过位错切割析出物机制实现。500°C 时效样品中析出物的 Ni/Al 比例最高，为 1.7。

采用高温纳米压痕和高温拉伸设备对固溶态样品进行了测试，研究结果表明材料在 500°C 以下变形过程中，析出物的强化作用大于基体的热力学软化，材料通过位错的扩散进行变形；而 500°C 以上材料的空间激活体积迅速增大，析出物的粗化与新的位错系统的开动导致材料通过位错攀移进行变形，表现为材料的热力学软化。另一方面，材料在高温下的纳米压痕测试与极限拉伸强度存在良好的线性对应关系： $UTS = 0.212H + 127.92$ 。

最后，通过与当前的各类抗辐照合金钢进行对比，讨论了 PH13-8Mo 在核工业领域中的应用前景以及采用机械热处理方法进一步提高 PH13-8Mo 性能，拓宽其应用范围

的可能。

关键词：辐照硬化、位错变形、微-宏观转换

厦门大学博硕士论文摘要库

Abstract

Mar-aging stainless steel is widely used in the fields of aerospace and energy applications due to its excellent mechanical properties and stable behavior under high temperature conditions. Owing to its well-balanced thermal expansion coefficient, pronounced anti-corrosion and -oxidation properties as well as pre-aged good machinability, it has caught the attention from the nuclear industries for a long time. In addition, high density small dispersive NiAl can precipitate in the martensitic after aging, which potentially provide traps for the irradiation individual voids, making the material radiation-tolerant and a strong candidate for Generation IV reactors.

In this thesis, mar-aging steel PH13-8Mo was selected for study and the main focus was on its mechanical and microstructural changes after long-term aging. After 2000 hours heat treatment at 450, 500 and 600°C, X-ray diffraction (XRD) and scanning electron microscope were utilized to characterize the phase and microstructure in the aged specimens. Aging at 450°C, it was found that starting from a solid-solution state, the steel maintains its properties after reaching peak hardness. On the contrary, in 500 and 600°C aged specimens; the coarsening of precipitates and the increasing reverted-austenite was believed to be in charge for the hardness degradation of the steel.

A linear accelerator facility was used to introduce 1.5 MeV protons to bombard three specimens, including the solid-solution state as well as the 500 and 600°C 2000h-aged-state ones at 300°C. The accumulated irradiation damage reached 5 dpa. Nanoindentation was performed on these samples and pronounced size effect was found in the 450°C sample. Irradiation hardening effect in the 500°C specimen was not as much as those in the other two.

To study the dynamic mechanical properties at elevated temperatures, in-situ high temperature nanoindentation as well as high-temperature tensile test were performed on the solid-solution state steel. Under 500°C, it was found that the strengthening effect of precipitates were more pronounced than the softening of matrix in such circumstances. Dislocation diffusion was considered to be responsible for the deformation. Above 500°C the activation volume of the steel increased rapidly which is associated with the coarsening of precipitates, and dislocation climbing was believed to take place during the deformation. In addition, the expression between micro- and macro- mechanical property was evaluated to have good linear relationship: $UTS=0.212H+127.92$.

3D atom probe tomography was used and specimens in the irradiated and unirradiated areas of the 500°C and 600°C aged samples were reconstructed. The analysis proved that ion beam irradiation accelerated the formation of NiAl small precipitates in the 600°C samples, leading to a greater size and number density than that of the unirradiated specimens. Moreover, strengthening mechanism was studied and the Orowan mechanism was believed to take place in the 500°C sample while at 600°C the strengthening of steel was realized by the precipitate-dislocation cutting mechanism.

In the end, a comparison between PH13-8Mo and other irradiation resistance steels were made, and a further processing technique of thermal mechanical treatment was considered to be an effective way to strengthen PH13-8Mo and extend its applications in elevated environment.

Key words: irradiation hardening; size effect; dislocation deformation; micro- and macro- mechanical transformation.

Degree papers are in the “[Xiamen University Electronic Theses and Dissertations Database](#)”.

Fulltexts are available in the following ways:

1. If your library is a CALIS member libraries, please log on <http://etd.calis.edu.cn/> and submit requests online, or consult the interlibrary loan department in your library.
2. For users of non-CALIS member libraries, please mail to etd@xmu.edu.cn for delivery details.