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# 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

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

A Dissertation Submitted for the Degree of Doctor of Philosophy

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# 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、微一宏观转换

# 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.