氏　　　　名	LI YANFEN
学位（専攻分野）	博士（工学）
学位記番号	総研大1277号
学位授与の日付	平成２１年９月３０日
学位授与の要件	物理科学研究科　核融合科学専攻 学位規則第６条第１項該当
学位論文題目	Effects of thermal ageing on mechanical property and microstructural evolution for reduced activation ferritic/martensitic steels
論文審査委員	主　査　教授　今川　信作 准教授　増崎　貴 教授　幸野　豊（室蘭工業大学） 研究副主幹　芝　清之（日本原子力開発機構） 教授　室賀　健夫

論文内容の要旨

Blanket is one of the key components of fusion reactors, which provides the main heat transfer and tritium breeding systems. Currently, reduced activation ferritic/martensitic steels (RAFMs) are considered as the primary candidates for blanket structural materials because of their most matured technological base and good irradiation resistance. In the past two decades, several advanced RAFM steels have been developed with continuous improvements in the world. Among these, JLF-1 and CLAM steels are Japanese and Chinese candidates, respectively.

In fusion reactor application, the structural materials will be exposed to long-term loading at high temperature, which may result in the changes of mechanical properties and microstructure. The thermal changes of properties and microstructure at the operation temperature are called ageing. Thermal ageing may reduce the maximum operation temperature by reduction of the yield strength or creep strength. However, up to now, the understanding of the ageing effects is not sufficient. The objectives of this work are: (1) to investigate the thermal ageing effects on mechanical properties changes, including hardness, tensile and creep deformation, (2) to clarify mechanism of the mechanical property changes by correlation with microstructural evolution, and (3) to apply the experimental data obtained to the blanket design.

In this study, the JLF-1 (JOYO-2-HEAT) and CLAM (0603 HEAT) were used for comparison. The chemical compositions (in weight%) were 9.00 Cr, 1.98 W, 0.49 Mn, 0.20 V, 0.083 Ta, 0.09 C, and balance Fe for JLF-1, and 8.94 Cr, 1.45 W, 0.44 Mn, 0.19 V, 0.15 Ta, 0.13 C, and balance Fe for CLAM. Thus, CLAM has higher level of Ta and lower level of W than those of JLF-1. The heat treatments included the normalization (1323 K/60 minutes for JLF-1 and 1253 K/30 minutes for CLAM) and tempering (1053 K/60 minutes for JLF-1 and 1033 K/90 minutes for CLAM). By comparing the two alloys, the effects of the composition and the heat treatment were investigated.

The thermal ageing experiments were carried out at a temperature range from 823 to 973 K up to 2000 h and under high vacuum to avoid oxidation. The temperature of 823 K was chosen to simulate the typical upper limit temperature for fusion reactor, and 973 K was chosen to accelerate the thermal processes. The mechanical properties including the hardness, tensile and creep were tested. Microstructure evolution was examined by Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM) equipped with Energy Dispersive Spectrometer (EDS).

The results showed that, the hardness increased slightly after ageing at 823 K for 100 and 2000 h for the both steels, suggesting ageing-induced hardening. However, the hardness decreased at 873 K and above, indicating ageing-induced softening. No significant changes were detected in both yield strength and ultimate tensile strength after ageing at 823 K for 2000 h, while the strength decreased at higher temperatures.

The creep curves of the both steels, similar to other RAFM steels, exhibited three regions: the short primary creep, secondary creep which was a linear process with a minimum creep rate, and tertiary characterized by an increased creep rate with time. After ageing at 823 and 873 K for 2000 h, the creep properties improved, while, they degraded significantly after ageing at 973 K for 100 h.

JLF-1 and CLAM steels before ageing exhibited a mixture of martensitic lath and tempered martensitic structure with two types of precipitates, $M_{23}C_6$ and TaC. Microstructural evolution revealed that, the number density of small precipitates (< 80 nm) increased significantly after ageing from 823 to 923 K up to 2000 h. However, after ageing at 973 K for 100 h, the density of small precipitates decreased, and the recovery partially of microstructure was also observed. By analysis of the chemical composition using EDS, most of the small precipitates were identified to be Ta-rich carbide (TaC), and the larger precipitates (~120 nm) to be the Cr-rich carbide ($M_{23}C_6$).

A traditional disperse obstacle model was used to correlate the hardness change and the microstructure. In this model, the dominant obstacle was assumed to be precipitates only. The results showed that the calculated hardness change almost agreed with the measured ones after ageing at 823 K for 2000 h, suggesting that the new formation of TaC was responsible for the hardening. However, there is a large difference between the calculated and measured dada after ageing at 973 K for 100 h, indicating that the loss of TaC alone cannot account for the softening. The recovery of lath structure and dislocations would contribute the major effects to the softening.

Comparing the two steels, the CLAM steel has higher hardness and tensile strength, smaller minimum creep rate and longer rupture time than those of JLF-1, while the susceptibility to thermal ageing of CLAM was larger than that of JLF-1. The lower normalization and tempering temperature and higher level of Ta were considered to be responsible for the finer grain and smaller martensitic lath width, thus leading to the higher hardness and strength for CLAM than those of JLF-1. However, the study showed that, because of the lower heat treatment temperature, the CLAM was more susceptive to thermal ageing, suggesting that the present heat treatment condition is not the best one. Increase in heat treatment temperature is necessary to improve the stability to thermal ageing for CLAM.

Since testing materials for the actual operation time is extremely costly and time-consuming, prediction of creep rupture performance in blanket conditions is critically important. In this work, the Larson-Miller parameter was used for prediction, which was based on the results of short-term creep experiments at higher temperatures with higher stresses. By prediction, the rupture stress at the typical blanket condition, 823 K for 100 000h, was estimated to be about 135 MPa for the both steels, and the present thermal ageing treatments influenced the rupture stress by about ± 15 MPa. This result can provide a reference for the design of fusion blanket.

In summary, this study has demonstrated the effects of thermal ageing on mechanical property and microstructural evolution for JLF-1 and CLAM steels. One of the important new findings is that the TaC is unstable, which can form newly or disappear by dissolution, during the ageing. Ta is an element newly used in RAFM steels, instead of Nb in the conventional steel for the purpose of avoiding long radioactivity. Thus limited understanding has been available of the status of Ta up to now. The present study showed that the future control of TaC can enhance the thermal properties of RAFM steels. Since the Larson-Miller parameter cannot incorporate the ageing effects, the pre-ageing experiment in this study can provide the suggestion for developing suitable methods to predict the creep performance in blanket conditions including the ageing effects.

博士論文の審査結果の要旨

核融合炉低放射化構造材料として早期の実用化に向けて開発が進められている「低放射化フェライト・マルテンサイト鋼（RAFM)」は、マルテンサイト変態を利用して高温強度を高めた材料であり、高温長期使用における強度の経時変化（時効効果）は重要な研究課題である。信頼できる時効効果の評価・予測には、長期高温保持による性能実証試験だけでなく、各強度特性間の相関、微細組織変化と強度特性の相関など原理的な研究を進める必要がある。低放射化フェライト鋼はその基本組成が確立してからの年月が短く、試験例も少ないので、限られたデータから長期間の材料挙動を予測する手法の高度化も必要である。

Li Yanfen 氏は、RAFM の時効効果の評価とその原理の探求、それに基づく材料と炉構造設計の高度化を目指した研究を行なった。日本と中国の RAFM 候補材である JLF-1（Ta 含有量：0.083%、熱処理：1050℃×60 分と 780℃×60 分）と CLAM（Ta 含有量：0.15%、熱処理：980℃×30 分と 760℃×90 分）を、550℃から 700℃まで、100-2000 時間の時効熱処理を行ない、硬度測定、引張特性試験、クリープ特性試験、透過電子顕微鏡組織観察を行った。その結果、RAFM の使用温度上限と想定される 550℃で 100-2000 時間保持することにより、硬度が上昇し、クリープ変形が減少することを示した。また組織観察により、この強度特性変化は高温保持により新たに TaC が析出したことが原因であることを見出した。TaC 析出は 650℃の保持温度まで起こり、マルテンサイト組織の回復による軟化との相殺効果を考慮することにより、得られた硬度、引張り特性、クリープ変形の保持温度・時間依存性が説明できることを示した。Ta は低放射化のため一般の耐熱フェライト鋼の Nb の代わりに新たに加えた元素であり、その挙動の理解は不十分であった。本研究は、TaC 析出の時効初期からの不安定性を初めて示すとともに、TaC を予めより多く析出させることによって、高温強度と熱的安定性の一層の向上が可能であることを示したもので、新規性の高い研究成果である。日中２つの候補材の比較においては、中国の候補材 CLAM が初期には優れた強度特性を示すものの、時効による性能劣化変化が大きく、この原因が熱処理温度・時間が不十分であったことに起因していることを突き止めた成果は、原型炉開発に向けて国際的に候補材の一本化を視野に入れた高度化を進めている現在、特に意義深い。また、本結果を用い、時効効果を考慮した核融合炉ブランケット条件での設計応力を導出していることは、研究成果の炉設計への積極的な活用と言う点で評価できる。さらに、時効効果の一部導入が不可避である熱クリープ試験において、予め時効を施した場合と比較することにより熱クリープ試験における時効効果を定量的に評価した成果は、熱クリープ挙動における時効効果の分離評価手法の確立に寄与する、学術性の高い研究である。

以上本研究は、現在の低放射化フェライト鋼材の基本的な問題を指摘し、その改善策を明示するとともに、ブランケット構造設計へ具体的に貢献するものである。さらに、熱ク

リープ挙動における時効効果の分離評価手法の確立に貢献し、学術的に価値のある研究である。以上により、本論文は学位論文として十分価値があると判断した。