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論 文 内 容 要 旨

1. Introduction

High Cr ferritic steels with a tempered martensite lath structure (subgrain) have been used in fossil fired power plants. Since their design lives are more than 10 years in fossil fired power plants, it is of engineering importance to understand their creep property in long-term range. However, it is not easy to carry out such a long-term creep test. The creep rupture life in long-term region is usually assessed by the extrapolation of short-term creep data. But this extrapolation often brings about overestimation of long-term creep rupture life because breakdown of creep strength takes place during long-term creep. The cause of this breakdown has not been fully understood yet. Therefore, the cause of breakdown is studied in the present study. In addition, the creep life of high Cr ferritic steels is assessed using hardness measurement.

2. Experimental procedure

The researched materials were two heats of Gr.91 steel. Creep tests were carried out under constant load in air. Before creep test, the steel was normalized and then tempered. After tempering, uniform tempered martensite lath structure was obtained. Hardness was measured with Vickers Hardness Testing machine under an applied load of 1.961 N. Subgrain width was evaluated using FE-TEM pictures. Spacing and chemical composition of precipitates were measured on the extraction replicas using EDS attached to FETEM-STEM.

3. Results

3.1. Cause of breakdown of creep strength

In the primary research, it has been pointed out that hardness drop of grip portion of crept specimens well accords with breakdown of creep strength during long-term creep. Based on this finding it is proposed that static recovery of tempered martensite lath structure is the cause of the breakdown. This proposal is assessed by studying the microstructural degradation of Gr.91 steel during aging, short-term creep and long-term creep, respectively.

3.1.1 Static recovery during aging

Both spacings of MX and $M_{23}C_6$ precipitates are kept constant in short-term aging. Spacing of MX precipitates is still kept constant in long-term aging, whereas spacing of $M_{23}C_6$ precipitates increases. Coarsening of subgrains takes place in long-term aging and is controlled by the aggregation of $M_{23}C_6$ precipitates, namely pure static recovery. The subgrain size controlled by aggregation of $M_{23}C_6$ precipitates is represented by the following equation:

$$d=k_2\lambda^2$$

where d is the subgrain size, k_2 is a constant (2.0×10^{-3} nm^{-1} at 700°C, 2.3×10^{-3} nm^{-1} at 600°C and 650°C), and λ is the spacing of $M_{23}C_6$ precipitates. The aggregation of $M_{23}C_6$ precipitates during aging is represented by the following equation:

$$\lambda^3=\lambda_0^3+k_1t$$

where λ and λ_0 are the spacing of $M_{23}C_6$ precipitates at the aging time t and before aging, respectively, k_1 is Ostwald ripening rate of $M_{23}C_6$ precipitates during aging.

3.1.2. Strain-induced recovery during short-term creep

Both spacings of MX and $M_{23}C_6$ precipitates are kept constant during interrupted short-term creep, whereas the subgrian size increases with increasing creep time. Coarsening of subgrains during interrupted short-term creep has nothing to do with MX and $M_{23}C_6$ precipitates and it is only caused by strain, namely strain-induced recovery. The correlation between subgrain size and strain during interrupted short-term creep is represented by the following equation:

$$d-d_0=k_6\varepsilon$$

where d and d_0 are the subgrain sizes during interrupted short-term creep and before creep test, respectively, k_6 is the coarsening rate of subgrains (3000nm), and ε is the strain.

3.1.3. Breakdown of creep strength during long-term creep

Aggregation of $M_{23}C_6$ precipitates takes place during interrupted long-term creep and it is larger than that due to pure static recovery, suggesting that strain accelerates the aggregation of $M_{23}C_6$ precipitates. Therefore, the aggregation $M_{23}C_6$ precipitates during interrupted long-term creep is caused not only by pure static recovery but also strain, namely strain-assisted static recovery. The aggregation of $M_{23}C_6$ precipitates during interrupted long-term creep is given by the following equation:

$$\lambda_{\text{Creep}} = k_7(\lambda_0^3+k_1\text{t})^{1/3} - (k_7-1)\lambda_0$$

where λ_{Creep} is the spacing of $M_{23}C_6$ precipitates at the creep time t, and k_7 is a constant (2.8 at 700°C and 40MPa, 5.3 at 700°C and 50MPa). The correlation between k_7 and creep condition should be studied more in future. Coarsening of subgrains during long-term creep is caused by strain-induced recovery, pure static recovery and strain-assisted static recovery. Their contributions are given by the following equations, respectively.

$$\Delta d_1 = k_6\varepsilon$$

$$\Delta d_2 = k_2(\lambda_0^3 + k_1 t)^{2/3} - k_2\lambda_0^2$$

$$\Delta d_3 = k_2[k_7(\lambda_0^3 + k_1 t)^{1/3} - (k_7 - 1)\lambda_0]^2 - k_2(\lambda_0^3 + k_1 t)^{2/3}$$

where Δd_1, Δd_2 and Δd_3 are the subgrain coarsening due to strain-induced recovery, pure static recovery and strain-assisted static recovery, respectively. The contributions of strain-induced recovery, pure static recovery and strain-assisted static recovery to the subgrain coarsening during long-term are additive. Contribution of static recovery including pure static recovery and strain-assisted recovery to the breakdown of creep strength is significant. This contribution is added to that due to strain-induced recovery, resulting in the breakdown of creep strength.

3.2. Assessment of creep rupture life using hardness measurement

The second objective in the present thesis is to assess creep rupture life of high Cr ferritic steels. The creep life is assessed using hardness measurement based on microstructural degradation during creep.

The correlation between hardness and subgrain size of the steel is given by the following equation:

$$H = k_4/d + B$$

where H is the hardness, k_4 is a constant (=3×10^4nm), d is the subgrain size, and B is a material constant (=141 in Gr.91 steel). The correlation between subgrain size and strain during interrupted short-term creep is given by

$$d - d_0 = k_6\varepsilon$$

Therefore, the correlation between hardness drop and strain during short-term creep is given by the following equation:

$$(H_0 - H)/[(H - B)(H_0 - B)] = k_6\varepsilon/k_4$$

where H and H_0 are the hardness during the interrupted short-term creep and before creep test, respectively. The short-term creep rupture life of the steel can be assessed by the following equation:

$$t_r^* = \frac{t}{1 - exp\{\Omega_2 \times [\varepsilon_0 + \frac{ln(1+\zeta t)}{\Omega_1} - \frac{k_4(H_0 - H)}{k_6(H - B)(H_0 - B)}]\}}$$

where ε_0 is the instantaneous strain upon loading, ζ, Ω_1 and Ω_2 are constants determining creep curve shape, and t_r^* is the predicted creep rupture life. The accuracy of short-term life assessment increases with increasing creep time. The accuracy of short-term life assessment is almost within 30% of the actual creep rupture life in the time range larger than 1/3 of creep life. This accuracy is good, so this hardness measurement method is applicable to the short-term life assessment of high Cr ferritic steels.

The subgrain size during interrupted long-term creep is given by the following equation:

$$d - d_0 = k_6'\varepsilon$$

where k_6' is dependent on creep conditions. It increases with decreasing creep stress. The value of k_6' is 1.2×10^4 nm at 700°C and 40MPa, and it is 8×10^3 nm at 700°C and 50MPa. Based on this equation and the correlation between hardness and subgrain size of the steel, the correlation between hardness and strain during long-term creep is given by the following equation:

$$\frac{(H_0 - H)}{(H - B)(H_0 - B)} = \frac{k_6'}{k_4}\varepsilon$$

The long-term creep life can be assessed based on the correlation between hardness and strain during long-term creep and the correlation between strain and long-term creep life which can be represented by creep curve. The accuracy of long-term life assessment is within 30% of the actual creep life in the creep time range larger than 1/3 of the creep life. This accuracy is good, so this hardness measurement method is also applicable to the long-term creep life assessment of high Cr ferritic steels. However, in order to use this method to assess long-term creep life, the creep condition dependent k_6' should be studied more in future. The long-term creep life is underestimated if the strain is assessed based on the correlation between hardness and strain during short-term creep. In addition, the underestimation decreases with increasing creep time.

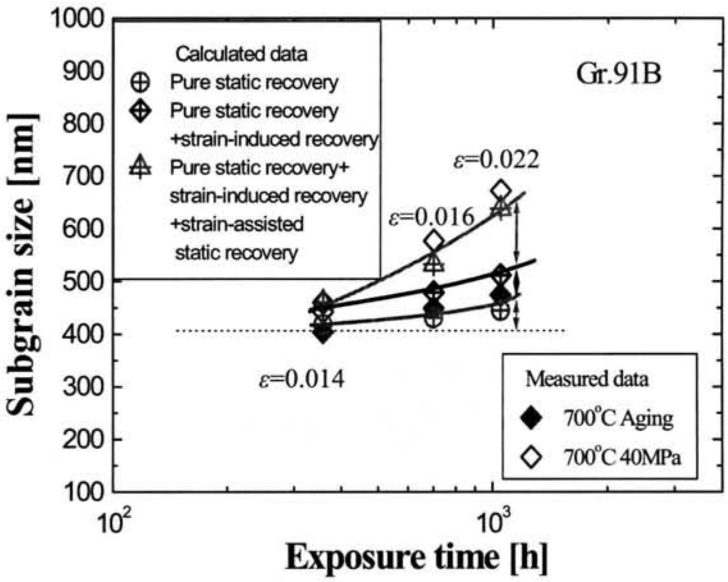

Figure 1. Subgrain coarsening of Gr.91 steel due to three types of recovery process

during interrupted long-term creep at 700°C and 40MPa

論文審査結果の要旨

蒸気火力発電プラントの高温部には，高 Cr フェライト鋼が数多く使われている。この種の耐熱鋼では，長時間使用後に，クリープ強度の急減が起きることが多い。日本の蒸気火力発電プラントの 80%は設計寿命を越えて使われており，これらの経年プラントでは，安全確保のために，構造部材の余寿命評価法の確立が求められている。このような背景のもとに本論文では，改良 9Cr-1Mo 鋼のクリープ損傷機構を明らかにし，この材料での強度急減の原因解明と寿命評価法の提案を行った。論文は全 8 章で構成されている。

第 1 章は序論で，本研究の背景を述べている。

第 2 章は目的で，研究の目的を述べている。

第 3 章は実験方法で，使用した材料，クリープ試験，組織観察の方法等を述べている。

第 4 章では，サブグレイン組織の回復が高温クリープ損傷の主原因であることを示すとともに，時効試験を行って，クリープ試験温度に暴露したことによるサブグレインの熱的回復挙動を研究した。その結果，$M_{23}C_6$ 炭化物粒子からのピン止め力の喪失がサブグレイン組織の熱的回復を支配していることを明らかにした。

第 5 章では， サブグレイン組織の熱的回復が起きない短時間クリープ条件で，変形に誘起されたサブグレイン組織の回復挙動を研究した。ひずみ誘起回復によるサブグレイン幅の増加量はひずみ量に比例し，比例係数はクリープ試験条件に依存しないことを明らかにした。

第 6 章では， サブグレイン組織の熱的回復とひずみ誘起回復が同時に起きる”長時間”試験条件で，サブグレイン組織の回復挙動を研究した。サブグレインのひずみ誘起回復しか起きない短時間クリープに対して，長時間クリープで熱的回復が加わることが，長時間クリープでの材料強度低下の原因であることを定量的に解明した。

第7章では， 主クリープ損傷であるサブグレイン組織の回復が硬さ測定で非破壊的に評価できることを提案し，硬さ変化を余寿命評価に適用する方法論を検討した。短時間クリープでは硬さ測定に基づく余寿命評価が有効なことを立証し，長時間クリープでは解決が必要な課題を明らかにした。

第8章は結論で，本研究の成果を総括している。

以上要するに，本論文は， 改良 9Cr-1Mo 鋼のクリープ損傷機構研究し，この材料でのクリープ強度急減の原因を解明し，余寿命評価法を提案したもので，環境科学の発展に寄与するところが少なくない。

よって，本論文は博士(学術)の学位論文として合格と認める。