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著者(和文)	高法剛
Author(English)	Fagang Gao
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Precipitation Kinetics and Microstructure Control of Fe_2Nb Laves Phase on Grain Boundaries in Fe-Cr-Ni-Nb Quaternary Austenitic Heat Resistant Steels

Nowadays, the research activities on the materials for the application on the 700ºC class advanced super-critical (A-USC) power plants have been conducted world widely. The present thesis focuses on the promising candidate material of Fe-Cr-Ni-Nb quaternary austenitic heat resistant steels strengthened by intermetallic phases of Fe_2Nb and Ni_3Nb targeting at the application at 1073 K, especially the precipitation control of the grain-boundary Fe_2Nb Laves phase in a system where both Fe_2Nb and Ni_3Nb phases need Nb atom for their formations. By identifying the Nb supersaturation for the precipitation of grain-boundary Fe_2Nb Laves phase as well as its effect on the equilibrium microstructure and precipitation kinetics, a novel steel is proposed to approach the ideal microstructure of 100% area fraction of Fe_2Nb phase on grain boundaries and only Ni_3Nb phase within grain interiors. The thesis outline is shown as follows:

In chapter 1 of "Generation Introduction", after describing the current state of the power generation in Japan, it is pointed out the materials with a 10^5 h creep rupture strength of over 100 MPa at 973 K are urgently required for the development of A-USC power plants for higher thermal efficiencies. Then excellent creep rupture strength of novel austenitic heat resistant steel Fe-20Cr-30Ni-2Nb (at.%, base steel) strengthened by intermetallic phases of Fe_2Nb and Ni_3Nb at 973 K is introduced, underlining the "*Grain-boundary precipitation strengthening* (GBPS)" mechanism by the Fe_2Nb phase and emphasizing the further application of the steel at 1073 K largely relies on the precipitation control of Fe_2Nb phase on grain boundaries in a system where both Fe_2Nb and Ni_3Nb phases need Nb atoms for their formation. Finally the significance and objective of the thesis are raised.

In chapter 2 of "Evaluation of Nb Supersaturation for the Precipitation of Grain-boundary Fe_2Nb Laves Phase", based on the study of γ+Fe_2Nb+Ni_3Nb three-phase co-existing region in Fe-Cr-Ni-Nb quaternary phase diagram, a model steel of Fe-20Cr-35Ni-2Nb (at.%) is proposed at 1073 K by maintaining the same molar fractions of both phases to that of the base steel at 973 K. The results clarify that the microstructure of the model steel nearly reaches to equilibrium state after 3600 h aging at 1073 K, where the Fe_2Nb phase precipitates on grain boundaries and within grain interiors and reaches to an area fraction of 60%, while the Ni_3Nb phase only precipitates within grain interiors. A method is proposed to evaluate the Nb supersaturation for the precipitation of grain-boundary Fe_2Nb phase, which is 0.35 at.% in the model steel, out of the total Nb supersaturation of 1.5 at.%.

In chapter 3 of "Change in Nb Supersaturation for the Area Fraction of Grain-boundary Fe_2Nb Laves Phase", the dependence of the Nb supersaturation for grain-boundary Fe_2Nb phase on the Nb and Ni contents is investigated with a Nb+ steel Fe-20Cr-35Ni-3Nb and a Ni+ steel Fe-20Cr-36Ni-2.5Nb (at.%), using the same method proposed in chapter 2. The results reveal that a 0.5 at.% Nb addition to the model steel increases the Nb supersaturation for grain-boundary Fe_2Nb phase by 0.2 at.% and raises the area fraction to 80%, while higher Ni content mainly increases volume fraction of the Ni_3Nb phase and shows little effect on the area fraction. Based on these results, a relationship between the area fraction and Nb supersaturation for grain-boundary Fe_2Nb phase is established, which suggests that a 0.85 at.% Nb supersaturation is required to approach to a 100% area fraction.

In chapter 4 of "Effect of Nb Supersaturation on the Precipitation Kinetics of Grain-boundary Fe_2Nb Laves Phase", the nucleation and growth behavior of both Fe_2Nb and Ni_3Nb phases as well as the effect of Nb supersaturation is investigated. The results show that Fe_2Nb phase precipitates first on grain boundaries in the model steel at 1073 K, followed by its precipitation within grain interiors, then the Ni_3Nb phase precipitates as meta-stable γ'', and transforms to stable δ at long-term aging. Nb addition accelerates the nucleation of grain-boundary Fe_2Nb phase, while Ni addition shows little effect, mainly enhancing the nucleation and growth of Ni_3Nb phase. Based on these results, a composition of Fe-20Cr-36Ni-3Nb (at.%) is proposed to enhance the grain-boundary precipitation of Fe_2Nb phase.

In chapter 5 of "Nucleation and Growth Kinetics of Grain-boundary Fe_2Nb Laves Phase through 3D Analysis", the different nucleation and growth behaviors of Fe_2Nb phase on grain boundaries is investigated by 3D observation. The results show that the nucleation kinetics of Fe_2Nb Laves phase is delayed on low-angle boundaries and twin boundaries, while two different types of behaviors appear on high angle boundaries: 1. fine granular particles of about 100 nm and high density, and holds for certain time; 2. larger granular particles of several 100 nm and low density, and grow along preferential growth direction. EBSD analyses reveal that the growth of Fe_2Nb phase tends to be faster when the grain boundary plane is close to the 111 plane of neighboring grains. The dependence of the morphology of Fe_2Nb phase on the grain boundary characters becomes weaker at long-term aging.

In chapter 6 of "Precipitation Control of Grain-boundary Fe_2Nb Laves Phase", a concept to control the precipitation of grain-boundary Fe_2Nb Laves phase is proposed based on the results of the former chapters. Ideally, an equilibrium microstructure should consist Fe_2Nb phase on grain boundaries with a 100% area fraction and only Ni_3Nb phase within grain interiors, and the precipitation sequence should be grain-boundary Fe_2Nb phase first, soon followed by the Ni_3Nb phase. Based on the effect of Nb supersaturation on the equilibrium microstructure and the precipitation kinetics, a composition of Fe-20Cr-37Ni-3.5Nb (at.%) is proposed.

In chapter 7 of "General Conclusion", the results and conclusions obtained in the thesis are summarized.