

論文 / 著書情報  
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題目(和文)	新たなFe-Cr-Ni-Nbオーステナイト系耐熱鋼の高温におけるFe <sub>2</sub> Nb(TCP)及びNi <sub>3</sub> Nb(GCP)相の析出と形態に及ぼす添加遷移金属元素の役割
Title(English)	Role of Additional Transition Element in the Precipitation and Morphology of Fe <sub>2</sub> Nb(TCP) and Ni <sub>3</sub> Nb(GCP) Phases in Novel Fe-Cr-Ni-Nb Austenitic Heat-resistant Steels at Elevated Temperatures
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**Role of Additional Transition Element in the Precipitation and Morphology of Fe<sub>2</sub>Nb (TCP) and Ni<sub>3</sub>Nb (GCP) Phases in Novel Fe-Cr-Ni-Nb Austenitic Heat-resistant Steels at Elevated Temperatures**

Recently, the microstructure design principle of novel austenitic heat resistant steels based on Fe-Cr-Ni-Nb system, strengthened by intermetallic phases of Fe<sub>2</sub>Nb Laves (TCP) phase on the grain boundaries, together with precipitation of Ni<sub>3</sub>Nb (GCP) phase within the grain interior, has been proposed. These design principle open possibilities to increase the temperature capability of Fe-base alloy above 1073 K and to develop the novel steels applicable for A-USC thermal power plants. However, in order to make further improvement of the creep strength, precise control of the supersaturation for the formation of the grain-boundary TCP phase (GBT), as well as the GCP phase in grain interior (GIG), is needed. In this study, thus, focuses are placed on the effect of additional element M on the microstructure control of GBT, from point views of thermodynamics and kinetics. The thesis outline is shown as follows:

In chapter 1 of “General Introduction”, in order to realize a low-carbon society and achieve stable supply of energy, it is urgently necessary to develop technologies for high efficiency thermal power generation, and to research the material applicable at higher temperature of 1073 K. The model steel of Fe-20Cr-35Ni-2.5Nb (at.%) strengthened shows superior creep strength at 1073 K, comparable to that of Ni-based alloys, through the “GBPS (grain boundary precipitation strengthening)” by the Fe<sub>2</sub>Nb-ε Laves (TCP) phase. However, in order to make further improvement of the creep strength, it is very important to construct the microstructure design principle for increasing the area fraction of TCP phase on grain boundary (GBT), which concerns the control independent control of the formation of the grain-boundary TCP phase, as well as the GCP phase in grain interior. In addition, it is pointed out that the addition of transition elements M (the 4<sup>th</sup> and 6<sup>th</sup> group elements in Periodic Table) is promising for independent control the precipitation of TCP and GCP phases. Finally, the significance, objective and structure of this study are explained.

In chapter 2 of “Effect of transition element (M: Ti, Mo, W) addition on the supersaturation for the precipitation of the TCP and GCP phases at 1073 K”, the effect of M on the supersaturation for the formation of grain boundary TCP phase to reach the area fraction ρ of more than 90 % is evaluated using the equilibrium microstructure of M added steels of Fe-20Cr-35Ni-2.5Nb (Base steel). Ti does not partition into TCP phase, while it increases the volume fraction of GCP phase, and it does not increase the area fraction. The 2 at.% Mo or W increases the area fraction from 60 % of base steel to 90 %, and W does not partition into GCP phase. Based on the evaluation of the supersaturation for the precipitation of the grain boundary TCP phase, it is found that W is the most effective element to increase the area fraction and an additional supersaturation of 0.23 at.% W is enough to increase the area fraction ρ to 90 %.

In chapter 3 of “Effect of Ti addition on the precipitation kinetics of the TCP and GCP phases in Fe-20Cr-35Ni-2.5Nb at elevated temperatures”, based on the supersaturation obtained from the chapter 2, the effect of Ti on the precipitation kinetics of TCP and GCP phases was examined, and the TTP diagram has been constructed. In the base steel, the precipitation of grain boundary TCP phase is first, followed by the precipitation and grain interior TCP phase, then the metastable GCP phase (Ni<sub>3</sub>Nb-γ") precipitates, following the transformation of the γ" phase to stable δ phase. The addition of the Ti enhances the nucleation of the grain boundary γ" phase and retards the nucleation of grain boundary TCP phase. Further addition of the Ti tremendously promotes the nucleation of the

metastable form of  $\gamma''$  phase both at grain boundaries and grain interiors. The metastable  $\gamma''$  phase transform to stable  $\delta$  phase through the discontinuous precipitation resulting in the rapid decrease in  $\rho$ , and the nucleation of  $\gamma''$  phase consumes the supersaturation for the formation of TCP phase.

In chapter 4 of “Discontinuous precipitation of the GCP  $\text{Ni}_3\text{Nb}$ - $\delta$  phase in Fe-20Cr-35Ni-2.5Nb-2Ti at 1073 K”, followed by the results in Chapter 3, the formation process of discontinuous precipitation of GCP was investigated. Discontinuous precipitation of GCP phase shows the finer lamellae with the moving of the grain boundary, then the fine lamellae change to the coarse lamellae by the discontinuous coarsening, and the interface of the lamellae is the nucleation site for the TCP phase. In addition, the area fraction rapidly decreases resulting from the mentioned microstructure change with the aging time.

In chapter 5 of “Effect of Mo addition on the precipitation kinetics of the TCP and GCP phases in Fe-20Cr-35Ni-2.5Nb at elevated temperatures”, based on the supersaturation obtained from the chapter 2, the effect of Mo on the precipitation of TCP and GCP phases was examined, and the TTP diagram has been constructed. The addition of Mo significantly promotes the nucleation of TCP phase on grain boundary by one order of magnitude and the  $\rho$  starts to increase in the nucleation stage. However, 3 at.% Mo addition changes the phase equilibria from three phases of  $\gamma$ +TCP+GCP to  $\gamma$ +TCP two phases, and the  $\rho$  does not change, owing to the nucleation of the non-equilibrium GCP phase with the higher supersaturation change to TCP phase resulting in the increase in the precipitation sites for grain interior TCP phase.

In chapter 6 of “Effect of W addition on the precipitation kinetics of the TCP and GCP phases in Fe-20Cr-35Ni-2.5Nb at elevated temperatures”, based on the supersaturation obtained from the chapter 2, the effect of W on the precipitation of TCP and GCP phases was examined, and the TTP diagram has been constructed. The W addition significantly promotes the nucleation of TCP phase on grain boundary more than Mo addition and also reflecting in more effect on the  $\rho$  start to increase in the nucleation stage. Similar with the Mo addition, 2 at.% W addition changes the phase equilibria from three phases of  $\gamma$ +TCP+GCP to  $\gamma$ +TCP two phases. 2 at.% W addition increases the  $\rho$  to 90% due to increase in the supersaturation for the formation of TCP phase on the grain boundary at nucleation stage.

In chapter 7 of “Microstructure control of the TCP and GCP phases in the novel austenitic heat-resistant steels by M addition at 1073 K”, based on the results from the thermodynamics (Chapter 2) and the kinetics (Chapter 3,4,5,6), a concept to control the precipitation of  $\text{Fe}_2\text{Nb}$  phase on the grain boundaries and  $\text{Ni}_3\text{Nb}$  phase within the grain interiors is summarized, and W and Ti addition is effective elements to control the TCP and GCP phase independently. Based on the area fraction for the unit atomic percent of added element and quantitative analysis for the change in the volume fraction of the grain interior GCP phase, the microstructure design principle for the proposed newly steel with excellent creep properties is proposed.

In chapter 8 of “General Conclusions”, the conclusions obtained from each chapter are summarized.