# Thermal Stability of 9Cr-1Mo Steels after Long Term Aging

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#### 1. Introduction

9-12% Cr-Mo ferritic/martensitic (FM) steels have been receiving attention for an application to the fuel cladding or reactor pressure vessel of various advanced nuclear reactors. Since the operating temperature and pressure of advanced nuclear reactors are supposed to be higher than those of light water reactors, high temperature mechanical properties and a microstructural stability of the cladding and reactor pressure vessel materials is very important. To understand the change of microstructure and mechanical properties during a thermal aging at elevated temperature is also important for the safety of an advanced nuclear power plant.

Therefore, an aging treatment was performed at 600 °C for up to 50,000 hrs and mechanical tests were carried out for the aged FM steels. And the effects of tungsten on the changes of microstructure and mechanical properties during a thermal aging were also investigated.

## 2. Experimental Procedure

The materials investigated in this study were 9Cr-1Mo and 9Cr-0.5Mo-2W FM steels. Two 30kg ingots were cast in a vacuum and hot rolled at 1150 °C into a 15 mm plate thickness. All the specimens were austenitized at 1050 °C for one hour and tempered at 750 °C for two hours. After an austenitizing and tempering, a thermal aging was conducted at 600 °C for 5,000, 10,000, and 50,000 hours.

The microstructural changes after a thermal aging were observed using a 200 kV TEM (Transmission Electron Microscope) equipped with an EDS (Energy Dispersive Spectrometer).

Fracture toughness of the aged specimens was investigated by a Charpy impact testing (three times each) at room temperature, by using a full-sized specimen containing a  $45^{\circ}$  V-notch. Vickers hardness measurements (ten times each) were conducted at room temperature and the averages were obtained. And the tensile properties were measured by using the Indentation-Typed Tensile Testing System (five times each).

# 3. Results and Discussion

## 3.1 Change of microstructure with aging

The microstructural changes with thermal aging treatments are usually the formation of new precipitates,

the coarsening of pre-existed precipitates, and the dissolution of pre-existed precipitates. Fig. 1 shows the TEM micrographs of the extraction replicas of the precipitates. The main microstructural change during a thermal aging at 600 °C was the formation of the Laves phase. The Laves phases are found around the  $M_{23}C_6$  precipitates on the prior austenite grain boundaries and the martensite lath boundaries [1]. The distribution and morphology of the Laves phase was similar among all the specimens. The Laves phase appears to be fairly larger particles, above 300nm in size compared to other precipitates. The size and number density of the Laves phase in the 9Cr-0.5Mo-2W steel were even larger than those in the 9Cr-1Mo steel.

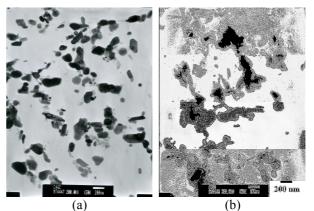


Fig. 1. Change of precipitates with aging time in 9Cr-1Mo steel (a) as tempered (b) aged for 50,000 hrs

## 3.2 Change of mechanical properties with aging

Fig. 2 shows the Vickers hardness change during a thermal aging. Laves phase is known to make the steel harder [2]. In the current study, the precipitation of the Laves phases occurred concurrently with the recovery of the dislocations and consequently the thermal aging did not really influence on the hardness.

The results of the indentation-typed tensile test have been compiled for the aging times of 5,000, 10,000, and 50,000 hrs. The effect of an aging on the room temperature tensile properties is shown in Fig. 3. The tensile test results show that the yield strength and the ultimate tensile strength were not significantly changed with an aging at 600 °C for 50,000 hours. Aging of the FM steels at the higher temperatures above 600 °C is reported to decrease the strength and to increase the ductility by a precipitate coarsening, extensive dislocation recovery, and a change in the subgrain morphology [3]. During a long term aging at 600 °C in this study, the precipitate was not largely coarsened and subgrains were not formed, only dislocation densities might be considerably decreased, so the strength of ferritic/martensitic steels appeared not to change much.

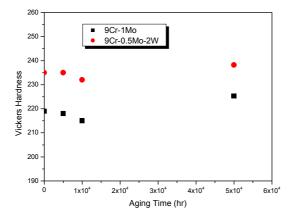


Fig. 2. Change of Vickers hardness with aging time

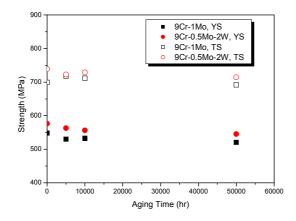


Fig. 3. Change of strength with aging time

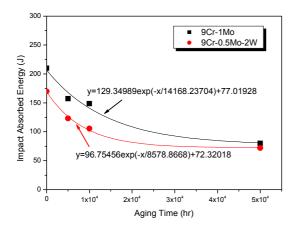


Fig. 4. Change of impact property with aging time

The results of the impact test at room temperature are shown in Fig. 4. The prior austenite grain size and the tempered strength are known to influence the impact property of the FM steels. The impact absorbed energy is reported to decrease with an increasing prior austenite grain size and strength [4]. In the as tempered state, 9Cr-1Mo steel had higher impact absorbed energy than 9Cr-0.5Mo-2W steel. The impact absorbed energy decreased with the aging time. The impact absorbed energy of the two steels exponentially decreased with an aging time. After 50,000 hrs of an aging at 600 °C, two steels reached a similar level of the impact absorbed energy (70-80 J).

It is known that the decrease of the impact toughness during an aging is due to a nucleation and growth of the carbide precipitates on the prior austenite grain boundaries and the martensite lath boundaries [5]. But in the present work the carbides were only slightly grown even after a long term aging at 600 °C. On the contrary a large amount of the Laves phases were formed after an aging for 5,000hrs in the 9Cr-0.5Mo-2W steel. The degradation of the impact property after a thermal aging at 600 °C for 5,000hrs is assumed to be mainly due to the formation of the Laves phase in the tungsten added steels. In the 9Cr-1Mo steel, however, the whole reduction of the impact absorbed energy is attributed to the growth of the precipitates and the lath width as well as the formation of the Laves phase.

#### 4. Conclusion

The microstructure of the FM steel was thermally very stable, so a coarsening of the precipitates was not significant during a thermal aging at 600 °C. The strength and hardness remained unaffected by a thermal aging at 600 °C up to 50,000hrs. But the impact absorbed energy decreased with the aging time in all the specimens.

#### REFERENCES

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