Abstract

Meeting the challenges of future energy demands will require nuclear power to play a significant role. Enhancing the safe operation of nuclear reactors is a high priority in the research community, especially in the materials sciences. Nuclear reactors require robust materials that can perform under the harsh conditions presented by a nuclear reactor. Many materials have been tested and experimentally verified for use in nuclear reactor systems with varying degrees of success. Two mechanisms have been often noted for their ability to enhance the properties of a material; refining grain size and the employment of nano-sized oxide particles.

F/M ODS steels have been the focus of much research in recent years with significant improvements noted over non-ODS steels. If the same improvements can be combined with the significant corrosion resistance improvement of austenitic steels, nuclear reactor operation safety and lifetime can be improved.

Chapter 1: Chapter 1 is an analysis of the world energy needs of today and in the future. Several different methods of energy production were briefly summarized and compared: coal, oil, natural gas, renewables, and nuclear power. Meeting future energy demands will require changes to all forms of energy production and nuclear power will play a significant role.

Chapter 2: In this chapter three main types nuclear structural materials were briefly summarized: zircaloy, ferritic/martensitic steels, and austenitic steels. The high temperature performance, corrosion resistance, and irradiation damage mechanisms were compared between each material type. Additionally, the strengthening mechanisms of refined grain size and precipitate hardening were discussed for each material.

Chapter 3: In this chapter, a mechanically alloyed austenitic stainless steel (MA304LZ) that was produced from pre-alloyed SUS304L powder with a small amount of Zr addition was analyzed. The yield stress (YS) of MA304LZ was 767 MPa at RT with a gradual decrease to 529 MPa at 500°C, indicating that MA304LZ YS is more than 3 times greater than that of SUS304L and SUS316L, while total elongation was reduced to about one third of the conventional steels. TEM/SEM Microstructure analysis revealed an average grain size of 0.42 μm in MA304LZ and about 34/30 μm in SUS304L/316L. In MA304LZ, two types of precipitates were observed; inhomogeneously distributed fine precipitates with an average size of 6.0 nm and homogeneously distributed coarse precipitates (d > 20nm) with an average size of 47 nm. The strengthening mechanism of MA304LZ was discussed on the bases of Hall-Petch and Orowan equations, and the strengthening of MA304LZ was attributed mostly

to refined grains. The dislocation barrier strength factor, α, is estimated to be 0.277 for the Zr-rich precipitates in MA304LZ.

Chapter 4: In this chapter, the precipitate behavior in same material as chapter 2 was analyzed. The precipitates had a bimodal size distribution causing strengthening of MA304LZ with 3.3 times larger yield stress than SUS304L, although the contribution of fine grains of MA304LZ to the strengthening was larger than the dispersion of precipitates. For precipitate characterization, two types of extraction replica samples were produced; 1) thin carbon film and 2) electrochemically dissolved residue. Extraction replica residue was analyzed using XRD and both types were attached to copper grids for TEM, EDS, and EELS analysis. The previously reported Zr-rich precipitates were revealed to be ZrO_2 (zirconia) with no evidence of ZrN or ZrC.

Chapter 5: In this chapter, the corrosion resistance properties of MA304LZ were analyzed. Coupon-type specimens of MA304LZ and SUS304L steels were subjected to hot water at 300ºC/25 MPa and supercritical pressurized water (SCW) at 500ºC/25 MPa for 1000 hr. MA304LZ is significantly less susceptible to corrosion weight gain in SCW than SUS304L which follows the parabolic rule between weight gain and elapsing time. The reduction of weight gain in MA304LZ can be attributed to much smaller grains which enhance chromium diffusion through grain boundaries and consequently accelerate the formation of a protective chromium oxide layer. Nickel oxides were observed in SUS304L but not in MA304LZ after the test at 500 ºC. It is considered that zirconium addition suppresses nickel diffusion as well as oxygen diffusion because of the strong interaction of zirconium/nickel and zirconium/oxygen. Electrochemical potentiodynamic reactivation (EPR) measurement of the degree of sensitization (DOS) of annealed and non-annealed samples indicates that both steels were resistant to sensitization of grain boundary corrosion. However, the annealing of MA304LZ at 1050ºC diminishes the beneficial grain size effect of MA304LZ.

Chapter 6: In this chapter, the effects of ion irradiation of MA304LZ, SUS304L and 316L were analyzed. Specimens of each material were irradiated with 6.4 MeV Fe^{3+} ions up to a nominal dose of 40dpa at 500ºC followed by FIB lift-out and TEM analysis for determining irradiation hardening and void swelling behavior. Nanonindentation testing was completed for comparing hardness before and after irradiation. Ion irradiation hardening was observed in all three materials, although the amount of hardening was at a minimum in MA304LZ. The amount of hardening for MA304LZ, SUS304L and SUS316L was 0.72 GPa, 2.51 GPa., and 1.23 GPa, respectively. Tangled dislocations and a high number density of dislocation loops

are observed in the damage region. Those complex dislocation structures are considered to be the main contributor for the irradiation hardening in each steel. The smallest hardening observed in MA304LZ can be attributed to the preferential absorption of interstitials atoms by grain boundaries of finely grained steel. Voids were observed in the damaged region excluding the peak damage region with the introduction of iron interstitial atoms. Remarkable void swelling regions were in the depth between 600 to 800 nm in all three materials and it varied among specimens with swelling for MA304LZ at 0.70%, SUS304L at 0.08%, and 316L at 0.48%. The largest void swelling in MA304LZ can be interpreted in terms of the preferential absorption of interstitial atoms by grain boundaries of the finely grained steel.
Chapter 7: This chapter is a summary of the contents of this doctoral dissertation.