ABSTRACT

Oxide dispersion strengthened reduced activation ferritic (ODS-RAF) steels are considered as promising structural materials for advanced generation nuclear reactors due to their excellent high-temperature mechanical strength and irradiation resistance. However, the ODS-RAF steels are still under development because of their poor plasticity and oxidation resistance.

The present work aims to understand the effect of silicon addition on the microstructure evolution, oxidation behavior, and mechanical properties of the ODS-RAF steels synthesized via mechanical alloying and spark plasma sintering followed by hot forging. The ODS-RAF steel powders of two different nominal compositions, Fe-14Cr-2W-0.3Ti-0.3Y_2O_3 and Fe-14Cr-2W-0.3Ti-0.3Y_2O_3-1Si, wt.%, were synthesized by mechanical alloying up to 50 h and consolidated via spark plasma sintering (SPS) at different sintering temperatures (900 °C, 1000 °C, and 1050 °C). An austenitic and martensitic transformation took place at all sintering temperatures in the Si-free ODS steel. However, in the case of Si-containing samples, this kind of phase transformation was observed only at higher sintering temperatures (≥ 1000 °C) owing to the presence of Si as a ferrite stabilizer. TEM analysis revealed the formation of $Y_2Ti_2O_7$ and Cr_2O_3 nano-sized particles in the Si-free ODS steel. On the contrary, the formation of Cr_2TiO_4 and SiO_2 along with $Y_2Ti_2O_7$ nanoparticles were observed in Si-containing ODS steel. Because of finer microstructure and absence of α-Fe to γ-Fe phase transformation, hardness and elastic modulus of the Si-containing ODS steel sample sintered at 900 °C were higher than that of the samples sintered at higher sintering temperatures.

After optimization of SPS temperature, samples consolidated at 900 °C (without forging) were selected for high-temperature oxidation at 850 °C for 100 h. Weight gain in the oxidized Si-containing sample (1.5 mg/cm^2) was ~ 18 times lower than that of the Si-free sample (27.5 mg/cm^2). Analyzing the surface and cross-section of the oxide layers via SEM-EDS, XRD, and Raman spectroscopy, it was revealed that the Si-free ODS steel consisted of outer Fe_2O_3 and inner $FeCr_2O_4$ layers which were porous and possessed whisker-like morphology. On the other hand, the oxidized Si-containing ODS steel possessed a strong protective outer layer of $(Fe,Cr)_2O_3$ and the inner layer consisted of a mixture of Cr_2O_3 and Fe_2SiO_4. Synergistic effect of the single crystal structure (only α-Fe) matrix and Si addition in the Si-containing ODS steel played a crucial role in forming thin, dense, and protective oxide layers during the oxidation process, resulting in improved oxidation resistance.

Succeeding to the optimization of SPS temperature and chemical composition, the Si-containing ODS ferritic steel made by sintering at 900 °C was forged at high-temperature to improve the densification and to avoid any influence of phase transformation (α-Fe to γ-Fe) and grain growth. Hot forging improved the densification from 95.6% to 98.2% and reduced the average grain size from 0.35 µm to 0.27 µm of the consolidated sample. The forged sample, as compared to the unforged counterpart (YS: 1710 MPa, CUS: 1795 MPa, TE: 21%, and hardness: 600 $HV_{0.3}$), exhibited 33%, 37%, 19%, and 13% improved yield strength, compressive ultimate strength, total elongation, and microhardness, respectively (YS: 2280 MPa, CUS: 2460 MPa, TE: 25%, and hardness: 680 $HV_{0.3}$). It was also observed that both compressive yield and ultimate strength of the forged sample decreased with increasing testing temperatures (25 °C, 500 °C, and 800 °C). A very good combination of strength and total elongation with the ductile mode of fracture of the forged sample at both room and elevated temperatures was obtained, which can be ascribed to good densification, finer grain size, and uniform distribution of nano-scale complex oxide particles ($Y_2Ti_2O_7$, Cr_2TiO_4, and SiO_2).

It was found that the Si-containing sample exhibited excellent oxidation resistance with favorable mechanical properties such as hardness, compressive strength, and plasticity. Thus, Si as a suitable alloying element in the ODS-RAF steel can be added for making low activation structural materials for the advanced generation nuclear reactors.

Keywords: Nanostructured oxide dispersion strengthened reduced activation ferritic steel; Silicon; Mechanical alloying; Spark plasma sintering; Microstructure; Oxidation behavior; Hot forging; Compression properties; Fracture behavior