ABSTRACT

With the onset of advanced gas-turbine technology, Ni-base superalloys started to replace Fe-base superalloys since they exhibited enhanced creep and fatigue properties. Ni-base superalloys owe their superior properties to the presence of ordered (γ′) precipitates. However, these precipitates coarsen during prolonged service at high temperatures, leading to a loss in strength. Cast and wrought Ni-base superalloys were also prone to grain coarsening and chemical inhomogeneity arising from segregation as well as due to the presence of oxides and carbonitrides. To overcome these difficulties, Powder-Metallurgy (P/M) route was used for preparing Oxide Dispersion Strengthened (ODS) Ni-base superalloys. In this class of alloys nano-sized yttria particles were added as dispersoids which contributed to a significant increase in creep strength. Nickel-base ODS alloys are extensively used as candidate materials for aircraft gas turbines including combustors, transition pieces, turbine vanes, blades and disks.

Most of the available literature lay emphasis on synthesis of Ni-base ODS alloys and evaluation of their high temperature properties along with the mechanisms involved. However, only limited literature is available on detailed microstructural analysis on high temperature consolidation of the alloy and their influence on the mechanical properties. Present research focuses on the synthesis of yttria dispersion strengthened Ni-base alloy by Mechanical Alloying (MA) and its subsequent consolidation using advanced compaction techniques such as Hot Isostatic Pressing (HIP) and Spark Plasma Sintering (SPS) followed by evaluation of physical and mechanical properties as well as its high temperature oxidation behaviour in detail.

Elemental metallic powder along with nano-crystalline yttria powders were mechanically alloyed using different ball mills viz. Simoloyer, Attritor and Planetary Ball Mill (PBM) to obtain Ni-base ODS alloy powders. Detailed characterization was taken up to study the microstructural evolution during the milling process. Optimization of process parameters for bulk production of ODS alloy powders were identified to be the best with PBM resulting in a crystallite size and lattice strain of 18 nm and 0.913 % respecively for a milling time of 40 h at 300 RPM. X-ray Diffraction (XRD) confirmed the formation of single phase FCC Nickel austenite phase. Scanning Electron Microscopy (SEM) analysis was done to study the progressive nature of milling during MA. Energy Dispersive X-Ray Spectroscopy (EDS) analysis confirmed the purity of the alloy powders. High Resolution Transmission Electron Microscopy (HRTEM) analysis revealed the presence of Moiré fringes and SAD pattern confirmed the formation of nano-sized grains in the Ni-base ODS alloy powders.

Mechanically alloyed powders were subsequently consolidated using Hot Isostatic Pressing at a temperature of 1453 K at 120 MPa for 3 h to give full density compacts of 99 % Theoretical density (TD). Optical images indicated a two-phase microstructure with matrix rich in Nickel austenite and chromium carbide as the second phase. The mean grain size and microhardness of the as-HIPed alloy were found to be 196 nm and 426.4 HV (at a load of 500 g) respectively. HRTEM analysis showed the bimodal grain size distribution in the HIPed sample. It was found that the size of yttria particles did not change even after subjecting to high consolidation temperatures during HIP indicating, no coarsening of the oxide particles. X-ray elemental mapping studies revealed the uniform distribution of the alloying elements in the HIP sample.

Mechanically alloyed Ni-ODS powders were also consolidated using the recent technique, Spark Plasma Sintering. Powders were spark plasma sintered under various conditions and density of the SPS samples was found to increase with an increase in sintering temperature. Samples sintered at 1373 K at 50 MPa, for 5 min resulted in maximum density of 98 % TD. Lattice strain calculated from XRD analysis was found to be 0.215 % which was higher than that of HIP samples. SPS samples showed a mean grain size of 135 nm and a microhardness of 514.6 HV (at a load of 500 g), which were significantly higher than HIP samples owing to lower sintering temperature and shorter times. HRTEM analysis showed the presence of annealing twins and a bimodal grain size distribution with coarse grains of 200 nm and fine grains of 60 nm. HRTEM images revealed the uniformly dispersed nano-sized yttria particles in the matrix alloy. Enhanced properties are achieved in SPSed samples because of the Ultrafine-Grained (UFG) microstructure and reduction in formation of chromium carbide precipitates, indicating the superiority of SPS process.

Isothermal oxidation studies were carried out on HIP and SPS samples at 1373 K for 25 h, 50 h, and 100 h. The kinetics of oxidation during 100 hour study was plotted in terms of weight gain as a function of time for HIP and SPS samples. The plot showed both transient and a steady state stage and the oxidation rate followed sub-parabolic growth behaviour. The transient stage was more evident in the case of HIP samples compared to the SPS samples. However, the oxidation rate of SPS samples was much smaller than HIP samples due to lower parabolic rate constant (k_p), indicating higher oxidation resistance. Fine microstructure obtained during SPS facilitated selective oxidation of Chromium to form an adherent chromium oxide (Cr_2O_3) layer on the surface of the substrate.