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Oxide dispersion strengthened nickel based alloys via spark plasma sintering

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ABSTRACT

Oxide dispersion strengthened (ODS) nickel based alloys were developed via mechanical milling and spark plasma sintering (SPS) of Ni–20Cr powder with additional dispersion of 1.2 wt% Y_2O_3 powder. Furthermore, 5 wt% Al_2O_3 was added to Ni–20Cr–1.2Y_2O_3 to provide composite strengthening in the ODS alloy. The effects of milling times, sintering temperature, and sintering dwell time were investigated on both mechanical properties and microstructural evolution. A high number of annealing twins was observed in the sintered microstructure for all the milling times. However, longer milling time contributed to improved hardness and narrower twin width in the consolidated alloys. Higher sintering temperature led to higher fraction of recrystallized grains, improved density and hardness. Adding 1.2 wt% Y_2O_3 to Ni–20Cr matrix significantly reduced the grain size due to dispersion strengthening effect of Y_2O_3 particles in controlling the grain boundary mobility and recrystallization phenomena. The strengthening mechanisms at room temperature were quantified based on both experimental and analytical calculations with a good agreement. A high compression yield stress obtained at 800 °C for Ni–20Cr–1.2Y_2O_3–5Al_2O_3 alloy was attributed to a combined effect of dispersion and composite strengthening.

1. Introduction

Increasing the operating temperatures in coal-fired power plants, gas turbine inlets, and other high temperature structural components in order to improve their efficiency and economy will require new materials with high mechanical and creep strength, oxidation and corrosion resistance. Nickel based alloys are promising candidates for such applications due to their excellent corrosion resistance at elevated temperatures [1–3].

While conventional nickel based alloys may not be very stable at high temperatures due to coarsening or dissolution of the second phase particles, nickel based oxide dispersion strengthened ODS alloys, reinforced by homogeneously dispersed nanoparticles (usually Y_2O_3), are quite stable during high temperature applications in excess of 1000 °C [4]. Homogeneous dispersion of nanometric stable oxide particles in the matrix of nickel based ODS alloys can act as effective barriers against dislocation motion [2,5–7] and improve high temperature mechanical properties including creep strength [8]. The pinning effects of oxide nanoparticles depend on the mean particle separation (the mean distance between the particles) which is a direct result of the particle number density [1]. According to theoretical calculations and experiments, a combination of a mean particle separation of 100–250 nm for 10–20 nm yttria particles and grain aspect ratio of a minimum of 10 could be promising for high-temperature applications [3,6].

In nickel based ODS powder containing both Al and Y_2O_3, different Y–Al–O particles such as $Y_3Al_5O_{12}$, $YAlO_3$ (perovskite), $Y_4Al_2O_9$ and $YAlO_3$ (hexagonal) can be formed during consolidation [1]. Recently, it has been noted that adding some minor elements such as Ti and Hf can replace the Y–Al–O particles with Y–Ti–Hf–O particles. The effects of adding minor elements such as Ti, Mg, Zr, Ca and Hf to Ni–0.5Al–1Y_2O_3 (wt%) were studied by Tang et al. [1], and Hf was found to be the most effective oxide at refining the formed oxide particles, especially at a concentration of 0.8 wt%. Formation of $Y_2Hf_2O_7$ was found to be responsible for oxide particle refinement and consequent improvement in mechanical properties through operation of the Orowan mechanism [9].

Another strengthening mechanism to be considered for developing nickel based ODS alloys would be composite strengthening mechanism or load transfer mechanism [10]. For example, studies have also shown that submicron Al_2O_3 of 0.5–1 μm diameter could be efficient for composite strengthening due to lower density and

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higher modulus of elasticity [11,12]. Hornbogen and Starke [13], and Rosler and Baker [14] predicted that a combination of nanoparticles and coarser particles dispersed in the microstructure would offer both dispersion strengthening and composite strengthening. Through dispersion strengthening and composite strengthening as dominant mechanisms at high temperatures, enhanced mechanical properties would be achieved.

Nickel based ODS alloys are conventionally produced by mechanical alloying (MA) or ball milling of elemental or pre-alloyed powders in combination with nano-sized Y_2O_3 (yttria) powder followed by canning, degassing and consolidation either via hot extrusion (for rods and wires) or hot isostatic pressing (HIP) and rolling (for sheets) [3].

One of the critical steps in producing nickel based ODS alloys is the milling process in which powder blends of yttria and pure nickel or pre-alloyed nickel (for example, Ni–20Cr) are milled, and a fine uniform distribution of yttria particles in the metal matrix can be attained. If powder blends of yttria and two or more metal powders are milled in addition to homogeneous yttria dispersion, formation of solid solution may be also achieved [3]. During milling, the metal powder particles become trapped between the colliding balls (milling media) and are cold welded together while the oxide particles become progressively finer until trapped between layers of metal powders sandwich forming a composite. After cold welding and particle agglomeration, a fracture stage occurs and large composite powders break down until a steady state situation is reached between cold welding and fracturing. Consequently, a uniform distribution of oxide nanoparticles within metallic components would be achieved [15,16].

In conventional consolidation methods such as extrusion or HIP, a final annealing at high temperature is usually required to develop a stable coarse grain structure [16]. The numbers of thermal processing steps could be eliminated if a pulsed direct current (DC) is simultaneously used with a uniaxial pressure to primarily sinter the powders [17,18]. This could reduce the time, cost and possibly deformation texture in the consolidated materials [19]. Field activated sintering technique (FAST), also known as spark plasma sintering (SPS) or pulsed electric current sintering (PECS), applies a pulsed DC to enhance sintering rate of the powders to near full density at relatively lower temperatures. Pulsed DC flows through the die and powder compact producing heat via Joule heating mechanism, providing a much higher heating rate and shorter sintering time compared to conventional sintering techniques. Thus, grain growth during sintering can be essentially minimized, leading to improvement in mechanical properties [18,20–22].

There are limited reported applications of SPS in the processing of nickel based ODS alloys containing Y_2O_3 as dispersoids [1,2,4,23,24]. Park et al. [2] developed Ni–22Cr–11Fe–1TiO_2, Ni–22Cr–11Fe–1Y_2O_3 and Ni–22Cr–11Fe–0.5TiO_2–0.5Y_2O_3 (wt%) by milling for 40 h in a planetary ball mill and SPSed the ball milled powder at 1100 °C for 5 min under a pressure of 40 MPa. They suggested that nano-sized TiO_2 and Y_2O_3 particles dissolved during MA, and then precipitated out during SPS, forming Y–Ti–O particles. However, Ni–22Cr–11Fe–1Y_2O_3 exhibited the best mechanical properties among all of the developed alloys.

In the present study, Ni–20Cr–1.2Y_2O_3 (wt%) alloy was processed by ball milling and SPS, and the effects of milling time and sintering parameters on the properties of sintered nickel based ODS alloy were investigated. There have been very limited studies on the effects of milling on the microstructural evolution during milling of nickel based powder [25]. Lopez et al. [25] milled elemental Ni and Cr powders for 30 h to obtain a nanostructured Ni–20Cr alloy. Such solid solutionizing reaction occurred mainly due to chemical-heterogeneity-driven diffusion through interfaces, subgrain boundaries and dislocation cores. In this study, Ni–20Cr and Ni–20Cr–1.2Y_2O_3–5Al_2O_3 (wt%) alloys were processed by milling and SPS, and their mechanical properties and microstructural evolution were studied in detail. Here, 1.2Y_2O_3 wt % (or 2 vol%) and 5Al_2O_3 wt% (or 10 vol%) were added to Ni–20Cr matrix for dispersion strengthening and composite strengthening, respectively.

2. Experimental

2.1. Powder processing and characterization

Gas atomized Ni–20Cr powder with nominal composition of Ni–19.6Cr–0.2Fe–0.8Mn–0.9Si (wt%) and mean particle size of 23.6 ± 1.1 μm; yttrium oxide (yttria/Y_2O_3) powder with high purity (99.99%) and mean particle size of 30–40 nm; and aluminum oxide (alumina/Al_2O_3) powder with 99.99% purity and mean particle size of 300–400 nm were all procured from the American Elements Inc.

Powder batches were prepared in a glove box under high purity argon atmosphere and poured into hardened steel grinding vial (Spex 8001). In order to minimize powder agglomeration and cold welding during milling, 1 wt% stearic acid was added to the powder mix prior to the ball milling process as a process control agent (PCA). The ball milling was carried out in a Spex 8000M shaker mixer/mill using steel balls 5 mm in diameter and a ball to powder ratio (BPR) of 10:1 (the powder mass and the ball mass of each batch was 10 g and 100 g, respectively). A variety of Ni-based alloys altering in milling time (0 h, 2 h and 4 h) and nominal composition (Ni–20Cr, Ni–20Cr–1.2Y_2O_3, Ni–20Cr–1.2Y_2O_3–5Al_2O_3, wt%) were milled.

For preparing the Ni–20Cr–1.2Y_2O_3–5Al_2O_3 powder, Ni–20Cr–1.2Y_2O_3 was first milled for 2 h, and then 5 wt% Al_2O_3 powder was added to the milled Ni–20Cr–1.2Y_2O_3 alloy and subsequently ball milled to distribute all the Al_2O_3 particles homogeneously. In our former experiments, Al_2O_3 powder was only blended (i.e. milled without the steel balls) with the milled Ni–20Cr–1.2Y_2O_3 powder, and the results were unsatisfactory because all the Al_2O_3 powder particles were found to be mostly located on the prior particle boundaries after consolidation.

X-ray diffraction (XRD) experiments of the as-milled powders were performed using a Siemens 5000D diffractometer with Cu-Kα radiation. Modifications such as kα_2 Rachinger and background correction by Sonnerveld were applied to XRD patterns using the Powder-X software [26]. Lattice parameters, crystallite size and lattice strain were calculated based on the Nelson–Riley extrapolation [27] and Williamson–Hall (W–H) formula, respectively [28]. For the instrumental broadening correction, a fully annealed/unmilled Ni–20Cr powder sample was used as a standard.

The morphology and size distribution of the as-received powder batches and as-milled powder were analyzed using a Zeiss Supra 35 field-emission gun scanning electron microscope (FEG-SEM). The milled powders were hot mounted in phenolic powder and polished to 0.05 μm. The cross section of the hot mounted and polished milled powders was observed in back-scatter electron (BSE) mode in SEM. A SEM micrograph obtained from the as received Al_2O_3 powder is shown in Fig. 1 and a high angle annular dark field (HAADF) scanning transmission electron microscopy (STEM) micrograph is presented in Fig. 1b.

2.2. Spark plasma sintering

The ball milled powder was consolidated via SPS using a Dr. Sinter Lab SPS-515S machine (SPS Syntex Inc., Kanagawa, Japan) with maximum capacity of 30 kN and 1500 A. A Tri-Gemini cylindrical graphite die with an inner diameter of 12.7 mm and an outer diameter of 38 mm was used. The inner surface of the die