



On the origin of strengthening mechanisms in Ni-Mo alloys prepared via powder metallurgy



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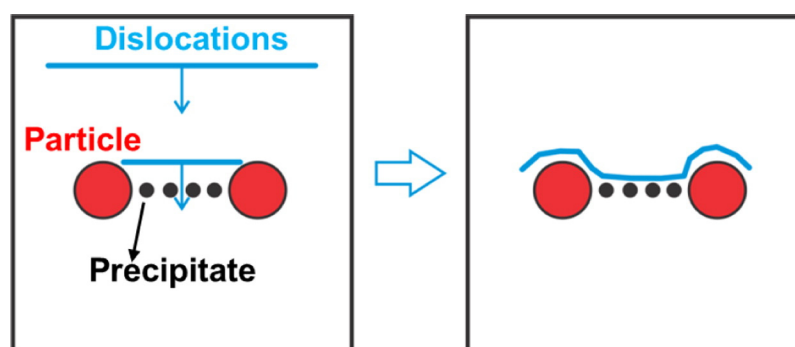
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HIGHLIGHTS

- A new design for a class of materials
- Combination of dispersion, precipitation and solid-solution strengthening
- Significant improvement of mechanical properties

GRAPHICAL ABSTRACT



A Ni-SiC composite is a class of the carbide dispersion strengthening (CDS) materials developed for the use in the future generation of Molten Salt Reactors (MSRs). However, it has been shown that the strength of this material is not satisfactory due to the large spacing between present SiC particles. Hence, in this work we designed a new class of materials, which can keep the advantages of SiC dispersion strengthening in addition to formation of nano-precipitates to further strengthen the material. A number of dispersion – precipitation strengthened (DPS) NiMo-based alloys containing varying amount of SiC (0.5–2.5 wt.%) was prepared via a mechanical alloying (MA) route followed by spark plasma sintering (SPS), rapid cooling, high-temperature annealing and water quenching. Lab X-ray Diffraction (XRD), Electron Back Scattering Diffraction (EBSD), and Transmission Electron Microscopy (TEM) were employed in the microstructural characterization. The Ni-Mo matrix of these new alloys is reinforced by dispersion strengthening of residual (unreacted) SiC particles from the initial powder mixture in addition to precipitation strengthening of nano-Ni₃Si precipitates, which precipitated during the sintering/annealing process. Furthermore, the matrix is strengthened by solid-solution of Mo in Ni. As a result, these newly developed NiMo-based DPS alloys relies on the combination of dispersion, precipitation and solid-solution strengthening leading to superior mechanical properties.

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ABSTRACT

A new class of materials, which rely on the dispersion strengthening of SiC particles in addition to precipitation strengthening by nano-precipitates is being developed for the application in molten salt nuclear reactors. A battery of dispersion and precipitation strengthened (DPS) NiMo-based alloys containing varying amount of SiC (0.5–2.5 wt.%) was prepared via a mechanical alloying (MA) route followed by spark plasma sintering (SPS), rapid cooling, high-temperature annealing and water quenching. Lab X-ray Diffraction (XRD), Electron Back Scattering Diffraction (EBSD), and Transmission Electron Microscopy (TEM) were employed in the microstructural

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characterization of this new type of alloys. It is shown that the NiMo matrix of these alloys is effectively reinforced by dispersion of SiC from the initial powder mixture and nano-Ni₃Si precipitates, which precipitated during the sintering/annealing process. Furthermore, the matrix is strengthened by solid-solution of Mo in Ni. As a result, these newly developed NiMo alloys take advantage of dispersion, precipitation and solid solution strengthening, which leads to their superior mechanical properties.

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1. Introduction

Molten Salt Reactors (MSRs) with advantageous features in inherent safety, economy, fuel cycle capabilities and a low primary loop pressure, has been selected as one of the advanced Generation IV concept reactors in 2002 [1,2]. Since the primary coolant of MSRs is FLiBe, a kind of corrosive molten fluoride salt, the structural materials used for the primary loop of MSRs will be subject to a great challenge of high-temperature strength, strong irradiation and corrosion [3]. The Ni-based alloy, such as Hastelloy-N, is considered as the candidate material in a number of MSR designs due to its outstanding corrosion resistance [4]. However, the weakness in high-temperature strength and irradiation resistance limits its application [5].

In recent years, carbide dispersion strengthening (CDS) materials have been studied due to the good corrosion resistance and high temperature stability of carbides [6,7]. In our previous studies, a SiC dispersion strengthening Ni-based (Ni-SiC) composite was successfully developed via a powder metallurgy (PM) route, see Ref. [8,9]. Microstructure characterization showed that SiC nanoparticles were well dispersed in the Ni matrix and possessed excellent thermal stability even at temperatures between 700 and 850 °C [10]. Furthermore, these carbide nanoparticles can effectively prevent the generation of He bubbles in the nickel matrix, thus greatly reducing the He embrittlement of the material [11,12]. However, the strength of Ni-SiC composite is not satisfactory for the application in MSRs, although a significant improvement after low-temperature annealing has been reported in Ref. [13]. This is mainly caused by the strengthening principle of CDS materials.

Unlike strengthening effect from oxide precipitates on the matrix of oxide dispersion-strengthened materials [14,15], carbide always keep stable as a separate phase in the matrix during milling and sintering [6,16]. Under the same volume fraction of carbide, the bigger grain size means a larger distance between the particles. Limited by the original grain size of carbide and grain growth during sintering, there is a big gap between two carbide particles, as shown in Fig. 1a. When dislocation lines moves through the gap, the short ones will pass through and the long ones will sharply bend and bypass the particles, thus missing obstacles. The pinning effect on dislocations largely decreases, resulting in the insignificant strengthening of the matrix. If some nano-precipitates can exist in the matrix at the same time, they can effectively act as obstacles to the dislocation motion, resulting in the significant improvement of strength, as shown in Fig. 1b.

In the present study, we designed a new class of materials, which can keep the advantages of SiC dispersion strengthening in addition to precipitation strengthening by nano-precipitates. Mo is chosen as an additive, because it can react with SiC at elevated temperatures [17] and dissolve into Ni matrix forming a solid-solution [18,19], thus further strengthening the alloy via solid-solution hardening. Hence, these newly developed NiMo alloys combine dispersion, precipitation and solid solution strengthening in order to obtain an alloy with superior mechanical properties. For the sake of this article we refer to these alloys as dispersion - precipitation strengthened (DPS) NiMo-based alloys.

2. Method

A battery of NiMo DPS alloy samples containing varying amount of SiC (0.5–2.5 wt.%) was prepared via a mechanical alloying (MA) route

followed by spark plasma sintering (SPS),¹ rapid cooling, high-temperature annealing and water quenching. A powder mixture containing 16 wt.% of Mo powder (99.6% of purity, 3.5 μm, bcc), 0.5; 1.0; 2; 2.5 wt.% of SiC (99.9% of purity, 30 nm, fcc), and the balance wt.% of Ni powder (99.6% of purity, 3.5 μm, fcc) was milled (MA) for 8 h in high-energy ball mill. The rotation speed of the mill table (disk) was set to 150 revolutions a minute, while four jars sitting on the mill table were rotating on its axis at the speed of 300 revolutions a minute. Prior to milling of the actual NiMo DPS powder mixture, the milling balls and jars were used to mill a NiMo powder mixture for 24 h in order to create a NiMo coating on the surface of the agate milling balls and jars. The left-over of this Ni and Mo powders was disposed before milling actual NiMo-wt.%SiC powder mixture. After the 8-hour milling process, the final NiMo-wt.%SiC powder mixture was consolidated using SPS process at 1100 °C under uniaxial pressure of 50 MPa employing a SPS furnace (KCE-FCT-HP D 25/4-SD). The mixture was pulse-heated² in vacuum within graphite die to 1100 °C (15 min), while the pressure was continuously increasing through graphite punch before reaching 50 MPa. After reaching desired temperature of 1100 °C and pressure of 50 MPa the powder mixture was sintered for 10 min, before being rapidly cooled via indirect water cooling system of pressing graphite punch. The final bulk density of all prepared alloys (NiMo-0.5SiC, NiMo-1SiC, NiMo-2SiC, NiMo-2.5SiC) in as-sintered condition reached about 98% of full theoretical density (measured by Archimedes method). With a view of producing stable precipitates within the microstructure, which would provide further precipitation strengthening of the Ni-Mo matrix, the prepared alloys in a form of as-sintered discs (diameter of 50 mm, thickness of 8 mm) were annealed at 1100 °C for 15 min and subsequently water quenched.

3. Results

3.1. Mechanical properties

The tensile mechanical properties of produced NiMo DPS alloys were obtained using dog-bone shaped samples at room temperature employing a Zwick Amsler 100 HFT 5100 tensile machine (0.001 s⁻¹). Fig. 2 presents the yield strength (YS), ultimate tensile strength (UTS), and total elongation (L) of prepared NiMo DPS alloys. For comparison we also present tensile mechanical properties of Ni-SiC composite from Ref. [8] alongside the NiMo DPS alloy results. Both YS and UTS of NiMo DPS alloys were found to be consistently higher than those of Ni-SiC composites, while showing significantly lower ductility. The results shown in Fig. 2a indicate that the YS of NiMo-based alloys is approximately 2 times, while UTS is approximately 1.5 times higher than that of Ni-SiC composites. Both YS and UTS of the NiMo DPS alloys decreased with an increasing amount of SiC content over 1 wt.% of SiC in the initial powder mixture. As regards the total elongation (L) shown in Fig. 2b, it becomes clear that ductility has reduced significantly with increasing amount of SiC in the initial powder mixture. The drop in the ductility is also significant in respect to Ni-SiC composites from Ref. [8].

¹ Also known as Pulsed Electric Current Sintering (PECS). The main characteristic of SPS is that the pulsed DC current directly passes through the graphite die, as well as the powder compact, in case of conductive samples, which results in achieving near theoretical density at lower sintering temperature compared to conventional sintering techniques.

² On/Off current. Pulse and pause time of current is 12 and 2 ms, respectively.

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