A study of densification and on factors affecting the density of Ni\textsubscript{x}–Fe\textsubscript{100–x} nanopowders prepared by mechanical alloying and sintered by spark plasma

Thanganadar Ashokkumar · Arunachalam Rajadurai · Gouthama · Linda L. Hussami

Received: 14 November 2011 / Accepted: 21 May 2012 / Published online: 6 June 2012 © Springer-Verlag London Limited 2012

Abstract Mechanical alloying through high-energy ball milling was used in the production of Ni–Fe alloy powders from elemental Ni and Fe powders of average particle size 80 and 25 μm, respectively. High-energy planetary ball milling at room temperature was performed for various time durations ranging between 2 and 100 h. SPS apparatus was used for sintering of powder particles. Density of all specimens was reported and a maximum densification of 99 % was achieved in 50 wt.% Ni–Fe milled for 16 h prior to spark plasma sintering at 1,223 K.

Keywords Fe–Ni alloy · Mechanical alloying · Particle size · Grain size · Spark plasma sintering · Densification

1 Introduction

From the manufacturing point of view, most of the existing techniques are still in an early stage of development and deal mainly with the manufacturing of nano- or micro-scaled products [1]. Initially, high-nickel (Ni) content alloys were used in submarine telegraph cables with an aim to increase the cable induction permitting a substantial increase in the signaling speed. Due to their unique properties, these alloys have become widely used in other fields of application, such as in audio frequency transformers and chokes, radio amplifiers, telephone repeaters as well as in magnetic shields. Permalloy and hypernic are hard magnetic alloys containing about 78 wt.% Ni–22 wt.% iron (Fe) and 50 wt.% Ni–50 wt.% Fe, respectively [2]. Miura et al. studied Fe–Ni-based metal–metalloid powders prepared by mechanical alloying and described that the amorphization of these systems (ductile to brittle) could be fully achieved by ball milling alone without performing any additional treatment, such as annealing [3]. Lately, ductile–ductile, ductile–brittle, and brittle–brittle powder mixtures were milled in the production of novel alloys. Mixtures of solid powder particles and liquids have also been milled in recent times [4]. Yavari et al. reported that mechanical milling is the most efficient method of mechanical alloying which may lead to complete solubility in the solid state of elements characterized by very low mutual solubility in the equilibrium condition [5]. Nanocomposites are multiphase materials fabricated from nanosized particles dispersed within a micron- or nanosized matrix or at the grain boundaries of the matrix, as described in the literature [6–8]. Processing nanoceramics inevitably has demanded the adoption of a specific processing route, which would restrict grain growth while achieving full densification. This became particularly feasible with the use of field activated sintering technique (FAST) involving the imposition of an electrical field during sintering. Spark plasma sintering (SPS) is one of the most widely used FAST. Zhang et al. synthesized fully dense bulk nanocrystalline Fe–0.8 wt.% C alloy by SPS of mechanically milled Fe–C nanocrystalline powder at 873 K [9]. The sintered sample was composed of 150 nm ferrite grains with nanocrystalline...
cementite dispersoids, whose compression yield strength, fracture strength, and plastic strain were 1,900 MPa, 3,500 MPa, and 40 %, respectively. Gopalan et al. reported that coercivities of 1,400 kA/m (17.6 kOe) and 650 kA/m (8.2 kOe) could be achieved in mechanically milled and spark plasma sintered Fe35Pt35P30 and Fe50Pt50 bulk magnets, respectively [10]. Detailed structural investigation was performed on WC–6 wt.% ZrO nanocomposites sintered for various time durations of up to 20 min at 1,573 K by Biswas et al. [11]. They suggested that effective sintering in SPS could also be obtained by applying 400 K less temperature than that of other conventional sintering methods. Transmission electron microscopy imaging revealed the structural characteristics of the particles formed during sintering: nanocrystalline ZrO2 particles (30–50 nm), relatively coarser ZrO2 particles (60–100 nm), submicron-sized WC grains as well as W2C. Kar et al. suggested that SPS and electro-discharge compaction technique could be used as an alternative method for sintering of nanomaterials and obtaining high degree of densification when pressure was simultaneously applied during the reaction [12]. In the present work, the effect of SPS on grain and particle size of Ni–Fe alloys; 40 wt.% Ni–60 wt.% Fe, 50 wt.% Ni–50 wt.% Fe, and 75 wt.% Ni–25 wt.% Fe is reported.

2 Experimental study

2.1 Materials

Powders of elemental Ni and Fe with a purity of 99.8 and 99.5 wt.% were used as starting materials.

2.2 Powder characterization

The as-received powders were analyzed for grain size, particle size, size distribution, and morphology. The average grain size of the initial Fe and Ni powders was calculated as 160 and 280 nm, respectively employing Scherrer’s equation [13]. The crystalline powders of Ni and Fe were analyzed for particle size distribution using Mastersizer 2000 particle size analyzer. The average particle size of Fe was 24 μm while that of Ni was 79 μm.

2.3 Powder preparation

Powder mixtures with initial constitutions of 40 wt.% Ni–60 wt.% Fe, 50 wt.% Ni–50 wt.% Fe, and 75 wt.% Ni–25 wt.% Fe (also written as 40 wt.% Ni–Fe, 50 wt.% Ni–Fe, and 75 wt.% Ni–Fe in the following) were selected as starting materials for the study. A single-pan balance with an accuracy of 1 mg was used for weighing the desired amounts of powders, which were subsequently mixed by hand in a ceramic bowl.

2.4 Synthesis

The Ni_x–Fe_{100–x} alloys were prepared by high energy planetary ball milling performed in a laboratory-scale planetary mill consisting of stainless steel jars and balls with a ball-to-powder weight ratio of 10:1 at a rotational speed of 300 rpm. The jars were sealed under ambient conditions and toluene was used as a process control agent. For studying the morphological, microstructural,