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CERAMICS INTERNATIONAL

Ceramics International 35 (2009) 3147-3151

www.elsevier.com/locate/ceramint

Rapid consolidation of nanocrystalline Al₂O₃ reinforced Ni–Fe composite from mechanically alloyed powders by high frequency induction heated sintering

Na-Ra Park^a, Dong-Mok Lee^a, In-Yong Ko^a, Jin-Kook Yoon^b, In-Jin Shon^{a,c,*}

^a Division of Advanced Materials Engineering and the Research Center of Industrial Technology, Engineering College,

Chonbuk National University, Chonbuk 561-756, Republic of Korea

^b Advanced Functional Materials Research Center, Korea Institute of Science and Technology, PO Box 131, Cheongryang, Seoul 130-650, Republic of Korea ^c Department of Hydrogen and Fuel Cells Engineering, Specialized Graduate School, Chonbuk National University, Chonbuk 561-756, Republic of Korea

> Received 18 February 2009; received in revised form 9 April 2009; accepted 3 May 2009 Available online 6 June 2009

Abstract

Nano-powders of Ni–Fe and Al_2O_3 were made from NiO and FeAl powders by high-energy ball milling. Nanocrystalline $5Ni_{0.6}Fe_{0.4}-Al_2O_3$ composite was consolidated by high frequency induction heated sintering (HFIHS) method within 2 min from mechanically alloyed powders of Al_2O_3 and Ni–Fe. The average grain size and mechanical properties of the composite were investigated. © 2009 Elsevier Ltd and Techna Group S.r.l. All rights reserved.

Keywords: B. Composite; C. Mechanical properties; Rapid sintering; Nanophase; NiFe-Al₂O₃

1. Introduction

The continuous increase in the performance requirement of materials for aerospace and automotive applications have lead to development of several structural composite materials. Among these, metal matrix composites refer to a kind of material in which rigid ceramic reinforcements are embedded in a ductile metal or alloy matrix. Metal matrix composites combine metallic properties (ductility and toughness) with ceramic characteristics (high strength and modulus), leading to greater strength in shear and compression and to higher service temperature capabilities. The attractive physical and mechanical properties that can be obtained with metal matrix composites, such as high specific modulus, strength-to-weigh ratio, fatigue strength, and temperature stability and wear resistance, have been documented extensively [1–5].

Traditionally, discontinuously reinforced metal matrix composites have been produced by several processing routes such as powder metallurgy, spray deposition, mechanical alloying, various casting techniques and SHS (self-propagating high temperature synthesis). One of all these techniques, highenergy ball milling and mechanical alloying of powder mixtures, were reported to be efficient techniques for the preparation of nanocrystalline metals and alloys, which is a combination of mechanical milling and chemical reactions [6].

Nanocrystalline materials have received much attention as advanced engineering materials with improved physical and mechanical properties [7,8]. As nanomaterials possess high strength, high hardness, excellent ductility and toughness, undoubtedly, more attention has been paid for the application of nanomaterials [9,10]. In recent days, nanocrystalline powders have been developed by the thermochemical and thermomechanical process named as the spray conversion process (SCP), co-precipitation and high-energy milling [11–13]. However, the grain size in sintered materials becomes much larger than that in pre-sintered powders due to a fast grain growth during conventional sintering process. Therefore, even though the initial particle size is less than 100 nm, the grain size increases rapidly up to 500 nm or larger during the conventional

^{*} Corresponding author at: Division of Advanced Materials Engineering and the Research Center of Industrial Technology, Engineering College, Chonbuk National University, Chonbuk 561-756, Republic of Korea. Tel.: +82 63 2381; fax: +82 63 270 2386.

E-mail address: ijshon@chonbuk.ac.kr (I.-J. Shon).

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sintering [14]. So, controlling grain growth during sintering is one of the keys to the commercial success of nanostructured materials. In this regard, the high frequency induction activated sintering method which can make dense materials within 2 min, has been shown to be effective in achieving this goal [15,16].

The purpose of this work is to produce dense nanocrystalline Al_2O_3 reinforced Ni–Fe composite within 2 min from mechanically alloyed powders by using this high frequency induction activated sintering method and to evaluate its mechanical properties (hardness and fracture toughness).

2. Experimental procedure

Powders of 99% NiO (-325 mesh, Alfa) and 99% pure FeAl ($<200 \mu$ m, Sinagigong, Inc) were used as a starting materials. 3NiO and 2FeAl powder mixtures were first milled in a highenergy ball mill, Pulverisette-5 planetary mill with 250 rpm and for 10 h. Tungsten carbide balls (8.5 mm in diameter) were used in a sealed cylindrical stainless steel vial under argon atmosphere. The weight ratio of ball-to-powder was 30:1. Milling resulted in a significant reduction of grain size. The grain sizes of Ni–Fe alloy and Al₂O₃ were calculated by C. Suryanarayana and M. Grant Norton's formula [17],

$$B_{\rm r}(B_{\rm crystalline} + B_{\rm strain})\cos\theta = \frac{k\lambda}{L} + \eta\sin\theta$$
(1)

where B_r is the full width at half-maximum (FWHM) of the diffraction peak after instrument correction; $B_{crystalline}$ and B_{strain} are FWHM caused by small grain size and internal stress, respectively; *k* is constant (with a value of 0.9); λ is wavelength of the X-ray radiation; *L* and η are grain size and internal strain, respectively; and θ is the Bragg angle. The parameters *B* and B_r follow Cauchy's form with the relation-



Fig. 1. Schematic diagram of the apparatus for high-frequency induction heated sintering.

ship: $B = B_r + B_s$, where *B* and B_s are FWHM of the broadened Bragg peaks and the standard sample's Bragg peaks, respectively.

After milling, the mixed powders were placed in a graphite die (outside diameter, 45 mm; inside diameter, 20 mm; height, 40 mm) and then introduced into the pulsed current activated sintering system made by Eltek in South Korea, shown schematically in Fig. 1. The four major stages in the synthesis are as follows. Stage 1: Evacuation of the system. Stage 2: Application of uniaxial pressure. Stage 3: Heating of sample by induced current. Stage 4: Cooling of sample. The process was carried out under a vacuum of 40 mTorr.

The relative densities of the synthesized sample measured by the Archimedes method are over 95% of the theoretical value.



Fig. 2. Scanning electron microscope images of raw materials and milled powder: (a) NiO, (b) FeAl and (c) milled powder.

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