Mechanical Synthesis and Rapid Consolidation of Nanocrystalline 3Fe_{0.67}Cr_{0.33}-Al_{2}O_{3} Composite by High Frequency Induction Heating

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Nanopowders of 3Fe_{0.67}Cr_{0.33} and Al_{2}O_{3} were synthesized from CrO_{3} and FeAl powders by high-energy ball milling. A highly dense nanocrystalline 3Fe_{0.67}Cr_{0.33}-Al_{2}O_{3} composite was consolidated by a high frequency induction heated sintering (HFIHS) method within three minutes from mechanically synthesized powders of Al_{2}O_{3} and 3Fe_{0.67}Cr_{0.33}. The average grain size and mechanical properties of the composite were investigated.

Keywords: nanostructured materials, sintering, mechanical alloying, mechanical properties, composite materials

1. INTRODUCTION

Numerous structural composite materials have been developed in efforts to meet continuously increasing performance requirements of materials employed in aerospace and automotive applications. Among these, metal matrix composites are materials 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 improved strength in shear and compression and to higher service temperature capabilities than conventional materials. The attractive physical and mechanical properties that can be obtained through the use of metal matrix composites, including high specific modulus, strength-to-weight ratio, fatigue strength, temperature stability, and wear resistance, have been extensively documented [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 self-propagating high temperature synthesis (SHS). Notably, high-energy ball milling and mechanical alloying of powder mixtures, which combines mechanical milling and chemical reactions, has been reported to be an efficient technique for the preparation of nanocrystalline metals and alloys [6].

Nanocrystalline materials have received much attention as advanced engineering materials with improved physical and mechanical properties [7,8]. Nanomaterials possess high strength, high hardness, and excellent ductility and toughness, and increasing attention has accordingly been paid to potential nanomaterial applications [9]. The grain sizes in sintered materials are much larger than those in pre-sintered powders due to rapid grain growth during conventional sintering processes. Therefore, controlling grain growth during sintering is one of the key factors to the commercial success of nanostructured materials. High frequency induction activated sintering methods, which can be used to quickly manufacture dense materials within 2 min, are effective for achieving this goal [10,11].

The purpose of this work is to fabricate a new nanopowder using high-energy ball milling and a dense nanocrystalline Al_{2}O_{3} reinforced Fe-Cr composite within three minutes from mechanically alloyed powders via a high frequency induction activated sintering method, and to evaluate its mechanical properties (hardness and fracture toughness) and grain size.

2. EXPERIMENTAL PROCEDURE

Powders of 99.99 % CrO_{3} (< 5 \mu m, Junsei Chemical Co.) and 99 % pure FeAl (< 200 \mu m, Sinagigong, Inc.) were
used as starting materials. CrO$_3$ and 2FeAl powder mixtures were first milled in a high-energy ball mill, a Pulverisette 5 planetary mill, at 250 rpm for 10 h. Tungsten carbide balls (8.5 mm in diameter) were used in a sealed cylindrical stainless steel vial under an argon atmosphere. The weight ratio of balls-to-powder was 30:1. Milling resulted in a significant reduction of grain size. The grain sizes of the Fe-Cr alloy and Al$_2$O$_3$ were calculated using Suryanarayana’s and Norton’s formula [12],

$$B_r (B_{\text{crystalline}} + B_{\text{strain}}) \cos \theta = k \frac{\lambda}{L} + \sin \theta$$

where $B_r$ is the full width at half-maximum (FWHM) of the diffraction peak after instrument correction; $B_{\text{crystalline}}$ and $B_{\text{strain}}$ are the FWHM caused by small grain size and internal stress, respectively; $k$ is a constant (with a value of 0.9); $\lambda$ is the wavelength of the X-ray radiation; $L$ and $\eta$ are grain size and internal strain, respectively; and $\theta$ is the Bragg angle. The parameters $B$ and $B_r$ follow Cauchy’s form with the relationship $B = B_r + B_s$, where $B$ and $B_r$ are the 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 a pulsed current activated sintering system (Eltek, 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 the sample by induced current; and Stage 4 - cooling of the sample. The process was carried out under a vacuum of 40 mtorr.

The relative densities of the sintered samples measured by the Archimedes method were over 96 % of the theoretical value. Microstructural information was obtained from product samples polished at room temperature. Compositional and microstructural analyses of the products were carried out through X-ray diffraction (XRD) and scanning electron microscopy (SEM) with an energy dispersive X-ray analysis (EDAX). Vickers hardness was measured by performing indentations at a load of 50 kg and a dwell time of 15 s on the synthesized samples.

3. RESULTS AND DISCUSSION

Figure 2 shows SEM images of the used raw materials and milled powders. FeAl and CrO$_3$ powders have an angular shape and round shape, respectively. The X-ray diffraction results for the high-energy ball milled powders are shown in Fig. 3(c). The reactant powders of CrO$_3$ and FeAl were not detected, but the products, Fe-Cr alloy and Al$_2$O$_3$, were detected. From the above results, the mechanical alloy is formed completely during the milling process. The net reaction can be considered to be a combination of the following two reactions:

$$\text{CrO}_3 + 2\text{Al} \rightarrow \text{Cr} + \text{Al}_2\text{O}_3 \quad (2)$$
$$\text{Cr} + 2\text{Fe} \rightarrow 3\text{Fe}_{0.67}\text{Cr}_{0.33} \quad (3)$$

Reaction Equation 2 is a well-known exothermic reaction for which the standard enthalpy of the reaction ranges from $-1379$ kJ to $-1311$ kJ over a temperature range of 700 °C (just above the melting temperature of Al, 660 °C) to 1800 °C (just below the melting point of Cr, 1863 °C).