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Magnetically-triggered heating of Fe–Al powders

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ABSTRACT

Powders of Fe and Al with a 3:2 atomic ratio were mechanically milled for either 1 or 2 h and subsequently subjected to an alternating magnetic field (AMF) to evaluate their heating. The microstructures of both the milled and heated powders were characterized using X-ray diffractometry, differential scanning calorimetry (DSC), scanning electron microscopy, and vibrating sample magnetometry. The results show that the milled powders consisted of fine intermixed Fe and Al layers. These were found to be susceptible to heating in an AMF, and underwent a significant heat output (21.3 kJ mol^{-1}) around 400 °C, corresponding to the reaction between the Fe and Al layers to form B2-structured FeAl. The temperature increase depended on the square of the field strength and the frequency of the AMF. By using a Kissinger analysis of the exothermic peaks obtained in a DSC at different heating rates, the activation energy for the formation of FeAl was found to be 67 ± 10 kJ mol^{-1}.

1. Introduction

Over the past two decades, iron aluminides have been of great attraction as promising candidates for a broad range of industrial applications, because of their excellent high temperature oxidation and corrosion resistance, reasonably low density, good intermediate-temperature mechanical properties and low material cost [1–4]. Fe–Al alloys, however, are prone to be brittle at ambient temperature and to be weak above 600 °C. Considerable efforts have been made to improve their ductility in the past years, such as particle-induced slip homogenization [5,6] and grain refinement [7,8].

Mechanical alloying (MA), owing to its comparatively low cost, flexible processing parameters and large variety of obtainable products, has become an extensively used technique for the fabrication of metastable alloys, such as nanocrystalline [9], quasicrystalline [10], and amorphous alloys [11] that can often not be produced by conventional metallurgical methods on an industrial scale. MA is a powder-processing technique, consisting of repeated cold welding, fracturing and re-welding of powders, in a dry, high energy ball mill that gives rise, through a micro-sandwich morphology, to the mixing of elemental powders and finally to alloy formation [12]. There have been several reports on the microstructural evolution and magnetic behaviors of mechanically-alloyed Fe–Al powders as a function of both milling time and compositional variation [13–16]. For example, Varin et al. [17], recently, investigated the non-magnetic/magnetic transformation in B2-structured FeAl alloys, cold-worked by ball-milling and ascribed the strong ferromagnetic behavior to the loss of long-range order (LRO) and substantial lattice expansion after large strains. Although previous studies [18–22] were mostly focused on the quasi-static magnetic properties of the Fe–Al alloys, the magnetic behavior in a dynamic magnetic field has rarely been reported.

Joining of materials, both similar and dissimilar materials, in an inexpensive way that does not greatly affect the properties of the materials to be joined, is of a great practical value. Reactive multilayer foils, such as Ni/Al foils, composed of alternating layers of materials that have a negative heat of mixing, have been used as local heat sources to melt braze or solder layers and thereby join components [23–25]. An alternating magnetic field (AMF), at the appropriate field strength and frequency has been widely examined for heating magnetic nanoparticles located in tumors for hyperthermia treatment [26–31].

The present work is a systematic study of the microstructures, magnetic properties and heating behavior of Fe–Al (3:2 atomic ratio) powders fabricated by mechanical milling and subsequently subjected to an AMF. The experimental findings are expected to form the basis of developing the milled Fe–Al powders as a new approach to joining using the AMF as a trigger to initiate the reaction between Fe an Al, which produces substantial heating.

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2. Experimental

Alloy powders with nominal composition Fe-40 at% Al were prepared by mechanical milling using a water-cooled Union Process Szegvari attritor at a rotation speed of ~400 rpm using 4.76 mm dia. 440C ultra-hard wear-resistant stainless steel spherical balls under an Ar atmosphere. Elemental powders with a purity of 99% and a particle size of less than 10 μm were used. The ball-to-powder ratio was 10:1. Heating tests of as-milled powders were performed in a home-built AMF heating system, in which both the frequency and magnetic peak field strength are controllable. The test system is described in detail in Baker et al. [26]. A temperature recording system utilizing a fiber optic probe (temperature range: 0–295 °C) was set up for estimating the heating efficiency of the powders.

The phases present in the as-milled and as-heated powders were determined using a Rigaku D/Max 500 X-ray diffractometer (XRD) utilizing Cu K_α radiation operated at 40 kV and 40 mA. Measurements were performed by step scanning 2θ from 10° to 120° with a 0.02° step size. A count time of 1 s per step was used, giving a total scan time of ~1.5 h. The diffraction peak angles, θ, for the heated FeAl were measured using the Rigaku's Jade 5 software package and the best value of the lattice parameter, a_o, was determined by plotting the lattice parameters calculated from each of the peaks, a, against the Nelson-Riley (N-R) function, $\frac{1}{2}(\cos^2\theta/\sin\theta + \cos^2\theta/\theta)$, and extrapolating to N-R = 0.

The microstructures and phase compositions were characterized using a FEI XL-30 field emission scanning electron microscope (SEM), equipped with an EDAX Li-drifted energy dispersive X-ray spectrometer (EDS). Both milled and heated powders were not only fixed on a cylindrical holder using conductive tape to examine their morphologies, but also mounted in a phenolic resin and polished to a mirror finish using 0.05 μm alumina powder for microstructural characterization.

The oxygen content of the powders after milling and after annealing was determined using a LECO TC500. The carbon, hydrogen and nitrogen contents of the powders after milling and after annealing were determined using a Perkin–Elmer 2400 Elemental Analyzer by Intertek QTI (Whitehouse, NJ).

The thermodynamic behavior was investigated using a Perkin Elmer DSC 7 differential scanning calorimeter (DSC), by heating from room temperature to 700 °C at a variety of heating rates under flowing argon. The quasi-static magnetic properties of the powders before and after heating in an AMF were characterized using a Lakeshore model 7300 vibrating sample magnetometer (VSM). The magnetic behaviors of the as-milled powders at different frequencies were examined using a B–H loop tracer (Shb Instrument Inc.). The B–H loops were collected over a range from −200 Oe to 200 Oe at frequencies of 0.1–10 Hz.

3. Results and discussion

3.1. Microstructural characterization

The morphologies of the powder particles before and after heating in an AMF with field strength of 160 Oe at a frequency of 400 kHz for 60 s are shown in Fig. 1. Flake-like particles were observed in un-heated powders, a typical feature of ball-milled powder [12], see Fig. 1a,b. After heating in the AMF, the particle size increased significantly and the particles became roughly spherical due to the agglomeration of the small flake-like particles (Fig. 1d). This may be attributed to the reaction and melting between particles during heating.

Backscattered electron (BSE) images of the powders and X-ray spectra from the phases present after milling for different times are shown in Fig. 2. After 1 h milling, an intermixed microstructure composed of white areas and grey areas (Fig. 2a), which are Fe and

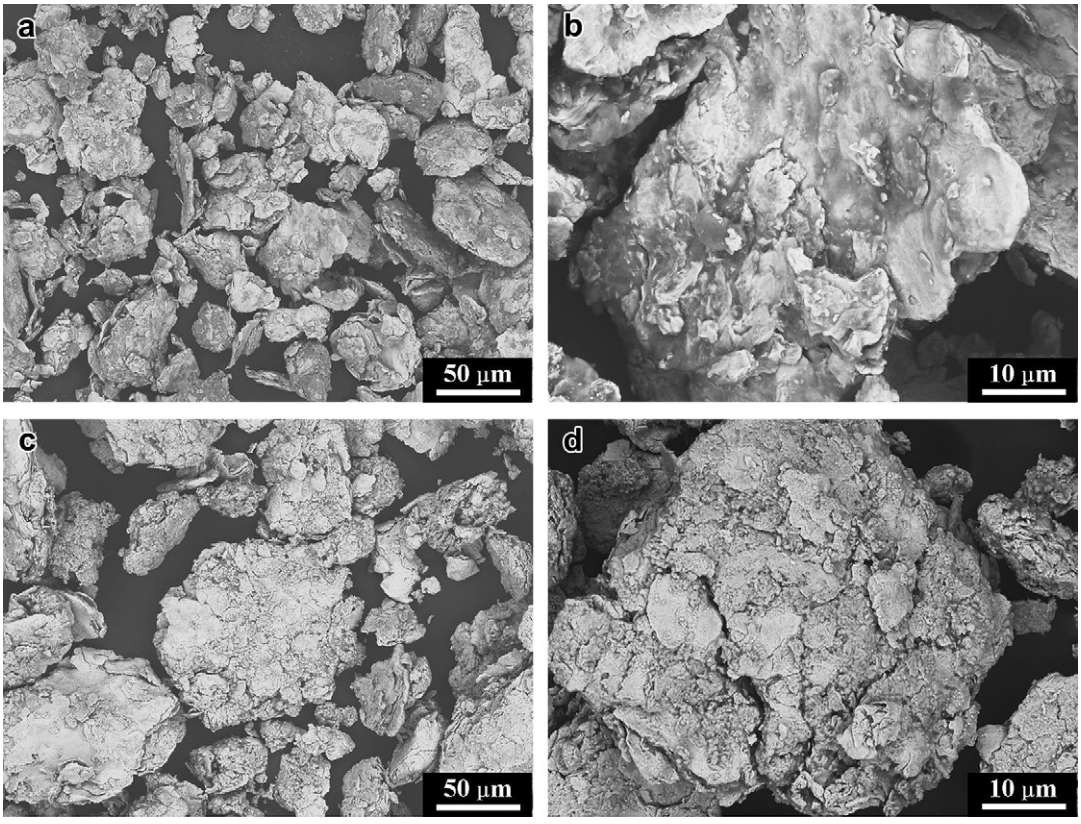

Fig. 1. Secondary electron images of the milled powder particles (a and b) before and (c and d) after heating in an AMF of 160 Oe at 400 kHz for 60 s.