Materials Science and Engineering A 527 (2010) 2313–2319

Contents lists available at ScienceDirect

Materials Science and Engineering A

journal homepage: www.elsevier.com/locate/msea

Effect of Mn element on microstructure and mechanical properties of bulk nanocrystalline Fe_3Al based materials prepared by aluminothermic reaction

Peiqing La*, Yupeng Wei, Ruijiao Lv, Yang Zhao, Yang Yang

State Key Laboratory of Gansu Advanced Non-ferrous Metal Materials, Lanzhou University of Technology, Lanzhou 730050, PR China

ARTICLE INFO

Article history:
Received 13 September 2009
Received in revised form
25 November 2009
Accepted 29 December 2009

Keywords:
Fe_3Al
Aluminothermic reaction
Mn element
Nanocrystalline
Yield strength

ABSTRACT

Bulk nanocrystalline Fe_3Al based materials containing 5, 10 and 15 wt.% Mn element were prepared by aluminothermic reaction. The materials were analyzed by X-ray diffraction (XRD), transmission electron microscope (TEM) and electron probe microanalyzer (EPMA). Compressive behavior and hardness of the materials were investigated. It shows that all of the three materials consist of nanocrystalline matrix and a little contamination phases. The nanocrystalline matrix phases are solid solutions composed of Fe, Al and Mn elements. The nanocrystalline matrix phases of the materials containing 5 and 10 wt.% Mn have disordered solid solution of bcc crystal structure and that of the material containing 15 wt.% Mn has ordered DO_3 crystal structure. Average grain size of the nanocrystalline matrix phases dramatically decreases at first and then increases a little with content of Mn and that of the material containing 10 wt.% Mn is the smallest. The material containing 10 wt.% Mn does not fracture in the compression and exhibits good plasticity, the yield strength and hardness increases at first and then decreases with content of Mn and those of the material containing 10 wt.% Mn are the highest.

1. Introduction

Intermetallic compounds have been intensively researched and developed because of their many unique properties [1,2]. In recent years there has been extensively interest in the development of iron-aluminide intermetallic such as FeAl, Fe_3Al, Al_3Fe as structural materials. Properties such as excellent resistance to oxidization and corrosion, low density and high elastic modulus make those materials apply to the aerospace industry such as compressor blades and missile skins, transportation, chemical industry and machinery [1–3]. Moreover, the main raw materials of iron-aluminide intermetallic are iron and aluminum, so they have price advantages compared to austenitic stainless steel and superalloys [1–3]. But the brittleness problem of Fe_3Al intermetallic compound severely hinders its wide application at room temperature [1–3]. Room temperature ductility of Fe_3Al can be increased by refining the grain size to nanometer, so recently considerable attention has been given to the development of nanocrystalline Fe_3Al [4–6]. Although considerable progress in basic understanding of nanocrystalline material has been made, it is much more difficult to obtain bulk nanocrystalline materials with large dimension for structural applications [7]. It has already been reported that the bulk nanocrystalline Fe_3Al based materials can be mostly produced by high velocity oxy-fuel (HVOF) [8], spark plasma sintering (SPS) [9], plasma activated sintering (PAS) [10]. In these methods, researchers prepared the nanocrystalline powders and subsequently consolidated them to gain the bulk nanocrystalline Fe_3Al based materials [8–10]. At the same time, the bulk nanocrystalline Fe_3Al based materials produced by SPS have the superior mechanical properties [9]. However, all of those methods are difficult to be scaled up to get large dimensions Fe_3Al based materials for applications in industry. In the last few years, aluminothermic reaction is developed as a convenient approach to bulk nanocrystalline Fe_3Al based materials preparation without the nanocrystalline powders as the precursor and has potential to scale up to get large dimensions Fe_3Al based materials [11].

Adding a certain amount of Mn element in microcrystalline Fe_3Al materials can improve room temperature ductility [12,13], while little research to add different content of Mn in bulk nanocrystalline Fe_3Al based materials is reported to date [14]. This paper is to investigate the effect of Mn element on microstructure and mechanical properties of the bulk nanocrystalline Fe_3Al based materials produced by aluminothermic reaction in order to tailor grain size and mechanical properties.

2. Experiments

Experimental raw materials are ferric sesquioxide, aluminum powders and Mn powders, their purities (wt.%) are all beyond 99%. There is a small amount of impurities such as Cl^-, SO_4^{2-}, N, Cu in

* Corresponding author. Tel.: +86 931 2976725; fax: +86 931 2976725.
E-mail address: pqla@lut.cn (P. La).

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doi:10.1016/j.msea.2009.12.043

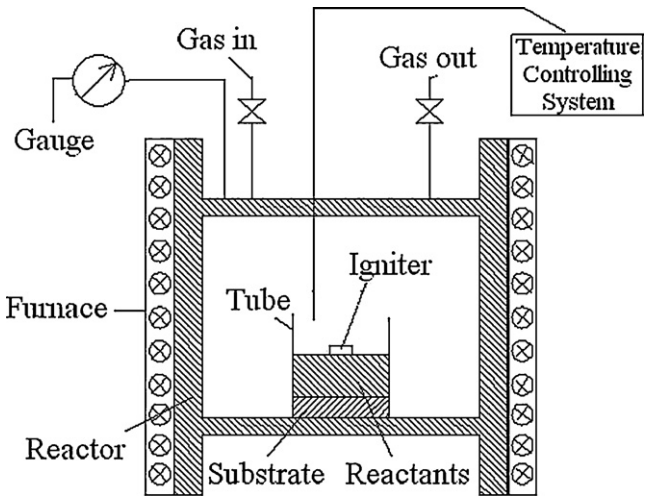

Fig. 1. Sketch of the aluminothermic reaction synthesis apparatus.

ferric sesquioxide powder and N, S, Fe, Cu in aluminum powder. Ferric sesquioxide and aluminum powders in terms of stoichiometry of aluminothermic reaction (Eq. (1)) and Mn powders that are 5, 10 and 15 wt.% in produced Fe_3Al were weighed and then dry-mixed for 16 h in a planetary ball mill where milling medium were Al_2O_3 ceramic ball, the mass ratio of ball to powders was 1:2 and milling speed was 150 rpm. Then 200 g of the mixed powders were placed on a copper substrate and pressed under a uniaxial pressure of 60 MPa in a copper tube. The tube and substrate were cleaned by ethanol before use.

$$3Fe_2O_3 + 8Al = 2Fe_3Al + 3Al_2O_3 \quad \Delta H^{\circ}_{f,298\,K} = -2550\,kJ\,mol^{-1} \quad (1)$$

Two grams of lamellar igniter was put on top of the pressed reactant powders and then the copper tube with the reactants was placed in a reactor as illustrated in Fig. 1. The reactor was purged with argon gas at room temperature and heated to temperature of 200 °C, and then purged for the second time. After 7 MPa argon gas was introduced, the reactor continued to be heated. Reaction of the igniter was started when the reactor was heated to temperature of 260 °C and exothermic heat was released from the igniter which ignited the reactants and caused the aluminothermic reaction of the reactants. The combustion waves of the reaction rapidly spread from top to bottom of the reactants and subsequently the reactants were transformed into Fe_3Al and Al_2O_3 products in area where combustion waves passed by. The products were melted by released heat of the aluminothermic reaction and in a superheated liquid state [11]. The synthesis reaction was finished in a few seconds and the products were kept in the reactor under argon gas pressure to cool. After cooling to room temperature, the products were taken out of the reactor. The gray Al_2O_3 product with little impurity was on top of the target products of the Fe_3Al based materials, which can be removed by hands. Using these above experimental procedures, bulk nanocrystalline Fe_3Al based materials containing 5, 10 and 15 wt.% Mn element were prepared. Those materials were about 80 mm in diameter and 8 mm in thickness.

Phases composition and microstructures of polished cross sections of the materials were examined by MeF3 optical microscope and EPMA-1600 electron probe microscope and D8 X-ray diffraction (XRD). The specimens were cut from the cross sections of the materials and ground into thin foils with less than 100 μm in thickness by hands, and subsequently punched into plates with a diameter of 3 mm by a puncher. The plates were thinned electrolytically by a twin-jet electropolishing device. The electrolyte

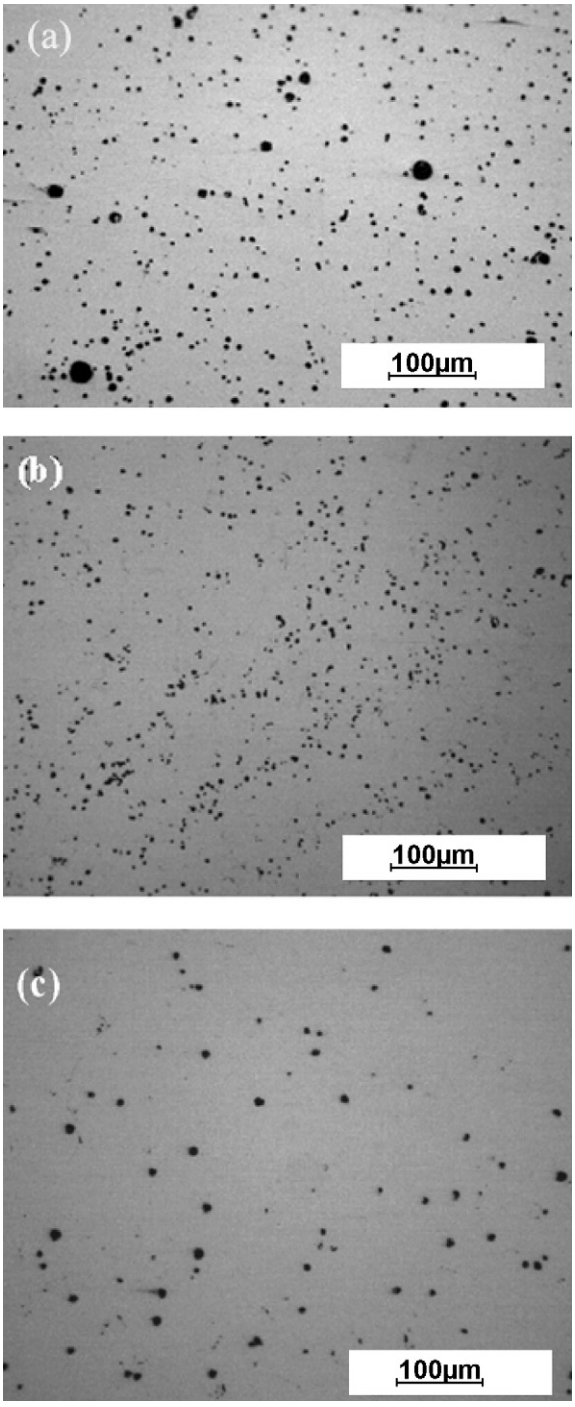

Fig. 2. OM images of the materials containing 5, 10 and 15 wt.% Mn element. (a) 5 wt.% Mn element; (b) 10 wt.% Mn element; (c) 15 wt.% Mn element.

was a 5 vol.% perchloric acid in ethanol solution. The plates were examined by JEM-2010 transmission electron microscope (TEM). Samples with 3 mm in diameter and 3 mm in height were cut from the materials by electro-discharge machining for compression and hardness tests. Compression tests were performed at room temperature using a Shimazu AT10t material mechanical properties tester with a crossing speed of 0.2 mm/min and repeated three times, the final value of yield strength $\sigma_{0.2}$ came from average value of the three tests. Hardness tests were performed using a HBRVU-187.5 tester under 298 N and repeated ten times, the final value of the hardness came from average value of the ten tests.