Journal of Alloys and Compounds 497 (2010) 90–94

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

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

Intermediary phases formation in Fe–Al–Si alloys during reactive sintering

Pavel Novák*, Vítězslav Knotek, Milena Voděrová, Jiří Kubásek, Jan Šerák, Alena Michalcová, Dalibor Vojtěch

Institute of Chemical Technology, Prague, Department of Metals and Corrosion Engineering, Technická 5, 166 28 Prague 6, Czech Republic

ARTICLE INFO

Article history:
Received 15 February 2010
Received in revised form 27 February 2010
Accepted 1 March 2010
Available online 6 March 2010

Keywords:
Reactive sintering
Powder metallurgy
Iron aluminide
Iron silicide
Wear resistance

ABSTRACT

Formation of intermediary phases during reactive sintering of Fe–Al–Si powder mixtures at 1100 °C was studied in this work. Results show significant difference from already presented results achieved at lower sintering temperatures. Phase composition is closer to the equilibrium phase diagram. Silicon additions up to 10 wt.% produce binary Fe–Al phases, further increase of silicon content leads to the formation of FeSi silicide and $Al_2Fe_3Si_3$ ternary phase. Binary FeSi40 alloy contains FeSi and $FeSi_2$. Porosity is reduced by addition of silicon. The lowest porosity was achieved when 10 wt.% of silicon was added. Formation of binary Fe–Al phases is controlled by aluminium diffusion through the layer of intermetallics. In Fe–Al–Si system, the controlling process is the reaction producing ternary intermediary phases. The highest wear resistance was determined in FeAl30Si10 alloy with single-phase FeAl structure. An increase of silicon content reduces the wear resistance although hardness increases.

1. Introduction

Reactive sintering is a densification process, where initial components in powder form are transformed into a compact product via thermally activated chemical reactions [1]. The route from powders to the compact usually concerns powder blending, cold pressing and sintering [2]. In some processes, cold pressing and reactive sintering are replaced by a reactive hot isostatic pressing [3]. The energy for the activation of chemical reactions during reactive sintering is supplied by heating in vacuum or protection-atmosphere furnace [2] or by electric discharge [4].

Reactions during reactive sintering production of intermetallic phases from metallic powders are strongly exothermal [5]. Evolved heat supports the propagation of reaction front through the material. In the case of aluminides, reactive sintering often produces highly porous materials [2]. By a pressureless reactive sintering production of FeAl alloy, relative density of maximum 75% can be obtained [6]. A promising solution of this problem with porosity consists either in pressure-assisted reactive sintering or alloying with other element, modifying the reaction mechanism. Previous results published in [7] revealed significant difference in phase composition and microstructure of FeAl20Si20 alloy developed in [8] between reactive sintering temperatures of 950 °C and 1100 °C. Phase composition of this alloy reactively sintered at 1100 °C was closer to the equilibrium ternary phase diagram published in [9]. In this work, reactive sintering was carried out at 1100 °C and silicon content varied in the same range as in our previous work [8] where the samples were sintered at 950 °C.

2. Experimental procedure

Fe–Al–Si alloys were prepared by reactive sintering of pressed powder mixtures of iron, AlSi30 master alloy, silicon (99.99%) and aluminium (99.99%). Aluminium and commercial AlSi30 master alloy powders with grain size of 200–600 μm were prepared by mechanical machining. Silicon powder with particle size <50 μm was obtained by mechanical milling. Iron was supplied in the form of p.a. purity powder with a particle size below 10 μm. Pressed powder mixtures (green bodies) were obtained by powders blending and uniaxial cold pressing under a pressure of 320 MPa using Heckert FPZ100/1 universal tensile-pressure loading machine, as optimized in our previous work [7]. Reactive sintering was carried out at 1100 °C in argon atmosphere with the heating rate of approximately 300 K min^{-1}. Such a high heating rate was achieved by placing the samples into the preheated electric resistance furnace. Microstructure of prepared materials was examined by Olympus PME3 light microscope. Philips X'Pert Pro X-ray diffractometer and Hitachi S-450 scanning electron microscope, equipped with EDS analyzer were employed to describe the phase composition. Hardness was measured by the Vickers method with the load of 5 kg. The abrasive wear resistance was evaluated by using a modification of the "pin-on-disc" method, where "pin" was the tested material and "disc" was a P1200 grinding paper. The applied load was 5.8 N and the sliding distance was defined as 2500 m. Wear rate was calculated from the measured weight losses by Eq. (1) [10]:

$$w = \frac{\Delta m \cdot 1000}{\rho \cdot l}, \quad (1)$$

where w, Δm, ρ and l are the wear rate (mm^3 m^{-1}), weight loss (g), density (g cm^{-3}) and sliding distance on the grinding paper (m), respectively. The density of samples was determined by the Archimedes method.

Experimental model system was utilized to describe the mechanism of the processes during reactive sintering. In this model, iron (AISI 1025 carbon steel) was submerged into molten AlSi30 alloy at 950 °C and 1100 °C. The reaction kinetics

* Corresponding author.
E-mail address: panovak@vscht.cz (P. Novák).

0925-8388/$ – see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.jallcom.2010.03.028

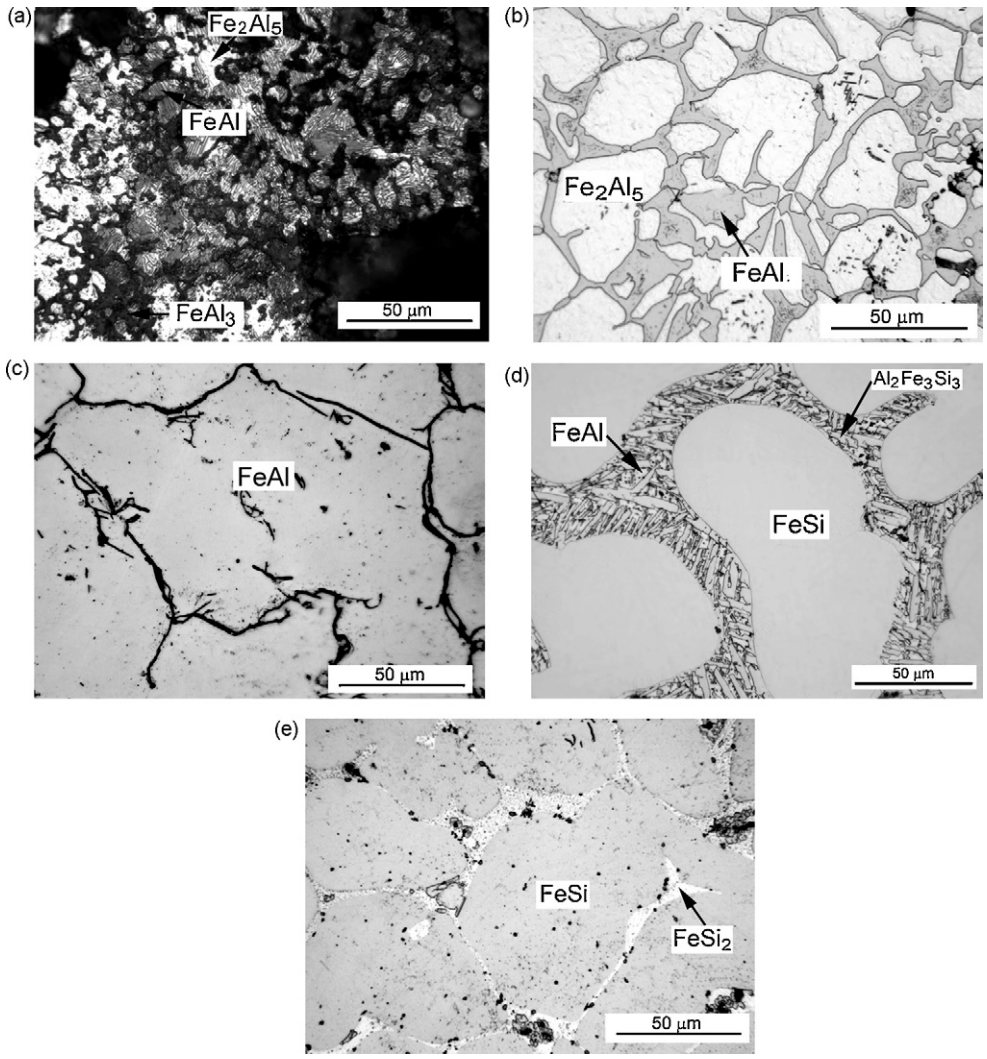

Fig. 1. Microstructure of Fe–Al, Fe–Al–Si and Fe–Si alloys produced by reactive sintering (1100 °C, 30 min): (a) FeAl40, (b) FeAl35Si5, (c) FeA30Si10, (d) FeAl20Si20 and (e) FeSi40.

was studied by measuring the thickness of the obtained layers by a light microscopy and image analysis (Lucia 4.8 image analysis software). The process controlling the formation of intermetallics was determined by fitting of the layer thickness by the linear or parabolic growth equation. Linear growth mode can be usually found during short reaction times. This so called linear law is described by Eq. (2) [11,12]:

$$d = K(t - \tau), \tag{2}$$

where d is the layer thickness, K is the linear rate constant, t and τ are the reaction time and incubation period, respectively. When the layer growth obeys the linear law, the process is controlled by a rate of the chemical reaction producing the intermetallics layer [11,12].

When a process is controlled by diffusion of species through a reaction product, it is generally described by the parabolic law, written as:

$$d^2 = D \cdot t, \tag{3}$$

where D is the diffusion coefficient of the most slowly diffusing element forming the layer [11,13].

Microstructure examination by the light microscopy was also applied to determine the sequence of intermediary phases formed by a reaction of iron with the melt. Intermediary phases were identified by scanning electron microscope (Hitachi S-450) equipped with Kevex Delta 5 EDS analyzer (SEM + EDS).

Table 1
Phase composition and porosity of Fe–Al–Si alloys produced by reactive sintering.

Alloy/reactive sintering temperature	Phase composition		Porosity (vol.%)	
	950 °C [8]	1100 °C	950 °C [8]	1100 °C
FeAl40	FeAl, Fe_2Al_5, $FeAl_3$	FeAl, Fe_2Al_5, Fe_3Al	36	39
FeAl35Si5	FeAl, Al_2FeSi, Al, Si	FeAl, Fe_2Al_5	7	7
FeAl30Si10	Al_2FeSi, FeAl, $Al_2Fe_3Si_3$, Al, Si	FeAl	3	9
FeAl25Si15	Al_2FeSi, FeAl, Al, Si	FeSi, FeAl, $Fe_2Al_3Si_3$	4	9
FeAl20Si20	Al_2FeSi, $Al_2Fe_3Si_3$	FeSi, FeAl, $Fe_2Al_3Si_3$	8	11
FeAl10Si30	$Al_2Fe_3Si_3$, FeSi	FeSi	35	15
FeSi40	Fe, Si, FeSi	FeSi, $FeSi_2$	38	17