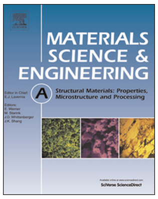
Materials Science & Engineering A 643 (2015) 72–85

Contents lists available at ScienceDirect

Materials Science & Engineering A

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



Microstructure evolution in a martensitic 430 stainless steel–Al metallic–intermetallic laminate (MIL) composite

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ARTICLE INFO

Article history:
Received 4 May 2015
Received in revised form
6 July 2015
Accepted 7 July 2015
Available online 9 July 2015

Keywords:
MIL composite
Metallic–intermetallic composites
Martensitic stainless steel
Microstructure evolution

ABSTRACT

The formation of intermetallic layers in 430-SS/Al MIL composites was investigated for reaction in solid/solid, solid/semi-solid and solid/liquid states. The Fe_2Al_5 phase was formed as a major phase in the solid-state reaction and co-existed with the τ phase ($Al_{5.5}Cr_{1.95}Fe_{2.55}$) as precipitates in a uniform layer in the semi-solid and liquid/solid reaction states. The growth of the uniform layer is mainly governed by lattice diffusion except for the grain boundary diffusion controlled mechanism at the early reaction stage. A porous two-phase layer consisting of Fe_4Al_{13} and δ phase (Cr_2Al_{13}) was formed in the semi-solid and liquid/solid reaction states and its growth is controlled by interface reaction growth when the liquid phase is present. The growth activation energies of the uniform layer and two-phase layer were calculated as 200 kJ mol^{-1} and 133 kJ mol^{-1}, respectively. A 430-SS/Al MIL composite with dense intermetallic layers was fabricated in the semi-solid reaction state, which was demonstrated to be the optimized process.

1. Introduction

Metal–intermetallic laminate (MIL) composites are hybrid laminate structures designed to optimize the desirable mechanical properties of intermetallics by incorporating layers of ductile reinforcement. The most widely researched MIL composites are based on the Ti–Al_3Ti system [1]. The microstructures of these MIL's can be designed to optimize the desirable characteristics of their constituent components, with the end result being a material that possesses the high strength and stiffness of the intermetallic phase and the high toughness and ductility of the residual metals. The composition, physical, and mechanical properties of the MIL composites can be varied and tailored within the thickness of the composite by simply varying the individual foil compositions, thickness, and Flayering sequence. The fabrication of MIL composites using this approach has several key advantages that make it ideally suited for the production of commercially scalable structural materials, as well as microstructures designed for specific functionalities. (1) Since the initial materials utilized are in the form of commercially available metallic foils, the initial material cost is reasonably low, compared to many of the exotic material processing routes that are commonly pursued in small-scale research environments. This also means that a wide array of compositions can be ready produced, although for this paper we will focus on the Fe–Al system because of their high specific properties. (2) The use of initially ductile metallic foils enables the layers to be formed into complex shapes. This opens the door for non-planar structures, such as rods, tubes, shafts, and cones, as well as simple machining of individual foils for complex, 3-dimensional structures, and near-net shape forming of parts. The use of initial metallic foils also allows the individual foils to be machined to contain cavities and pathways facilitating the incorporation of embedded functionalities, such as passive damping [1] or sensors, prior to processing. (3) The processing conditions, in terms of temperature, pressure and atmosphere are very modest. Processing temperatures, in the case of Al-foil containing samples are below 700 °C, and the processing pressures are below 4 MPa [2]. Perhaps the most remarkable feature of the processing of these metallic–intermetallic laminate composites is that the processing is carried out in open air, no special inert gas or vacuum chamber facilities are necessary. The combination of these various processing features makes the processing method itself very low cost, allows for complex shape fabrication and is easily amenable to computer control. (4) The microstructure of the metallic–intermetallic laminate composites is determined by the foil thickness and composition and the processing condition. Since the material make-up is based on the selection of the metal foils, it is possible to completely tailor the microstructure from one surface to the other. In addition, the physical and mechanical properties of the MIL composites can be tailored by selection of the foil

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http://dx.doi.org/10.1016/j.msea.2015.07.014
0921-5093/

composition and thickness making the MIL composite material system ideally suited for engineering the microstructure to achieve specific performance goals.

In the case of the Ti–Al_3Ti MIL composites, the evolution of the intermetallic phase in the microstructure has been studied by Harach and Vecchio [2], the fracture behavior has been detailed by Rohatgi et al. [3] and Adharapurapu et al. [4], and the fatigue crack resistance examined by Adharapurapu et al. [5]. Since much of the unique mechanical properties of Ti–Al_3Ti MIL composites has been shown to be derived from the hierarchical structure of the laminates and the significant degree of toughening achieved with even very small volume fractions of remnant metal layers, it is desirable to fabricate similar MIL composites from lower cost sheet metals, specifically replacement of Ti with an Fe-based sheet metal product. This paper examines the microstructure evolution in MIL composites fabricated from 430-martensitic stainless steel and commercially pure Al foils. Given that this stainless steel alloy contains at least 2 major elements (Fe and Cr) that both form aluminides, the phase evolution during the MIL processing is expected to produce more complicated intermetallic layers. One goal of this study is to examine whether processing these 430-SS/Al MIL composites in the solid-state, semi-solid state or via liquid–solid reaction yields an intermetallic layer that will optimize its mechanical properties.

The phase formation and grow kinetics between pure iron or steel with aluminum to form an intermetallic phase has been investigated at temperatures that are significantly below [6–8] or above [9] the melting point of aluminum. However, stainless steel alloys have not been systematically studied for their aluminide formation sequence or growth kinetics. Dybkov [11] studied the interaction of 306-stainless steel with liquid aluminum using the rotating disk technique. Two intermetallic layers were identified by EDS and XRD analyses: a compact layer adjacent to the steel surface as a compound of the type $(Fe,Cr,Ni)_2Al_5$, and a porous layer adjacent to the molten Al of the type $(Fe, Cr, Ni)_4Al_{13}$. Dybkov showed the effect of a chemical reaction step on the layer growth and experimentally confirmed linear growth kinetics after an initial parabolic one (paralinear growth kinetics). However, the exact types of intermetallic phases formed between 306-stainless steel react with Al, especially the Cr and Ni containing phases, have not yet been identified and confirmed utilizing advanced characterization techniques. Barmak et al. [12] has studied the dissolution process of iron–chromium alloys containing 10 and 25 wt% Cr in an aluminum melt at 700 °C. They also found two intermetallic layers: a uniform Fe_2Al_5 layer and a porous Fe_4Al_{13} layer with Cr_2Al_{13} and other phases existing as inclusions in the aluminum matrix. Barmak et al. have also studied the kinetic dissolution of Fe and Cr into the molten Al; however, little information about the kinetics and growth mechanism of stainless steel alloys reacting with Al and the associated interdiffusion processes of the components has been reported.

Most of the previous work on the reaction between Al and stainless steel was performed in the liquid Al state by hot dipping the stainless steel into molten Al [12,13]. There remains very limited experimental data on intermetallic growth in solid-state interdiffusion processes, which can be applied as a coating or bonding Al–SS bimetals [10]. Previous studies have shown that when Al reacts with other metals such as: Ti [2], Fe [14], Ni [15] and Cu [16], to form intermetallics, the type of intermetallic formed and the growth mechanism are strongly dependent on the reaction temperature. More specifically, whether the temperature is below or above the melting temperature of Al, separates the analysis of the reactions as solid-state reactions or solid–liquid reactions. However, the lowest eutectic temperature in the system was also seen as the critical temperature that effects intermetallic formation and kinetics [17]. Semi-solid processing, in which the alloy is in a two-phase (eutectic) "mushy zone" with one phase liquid and the other solid, has recently received significant attention in studies of low-carbon steel reacted with Al for its potential near-net shape processing [14]. In the present study, the reaction between Al and 430 SS has been separated into three temperature ranges: the solid-state, semi-solid state and the liquid reaction state.

2. Experimental

2.1. Materials and processing

Foils of commercial purity 1100-aluminum and 430-stainless steel (hereafter abbreviated 430-SS) were stacked in alternating layers and placed in a composite synthesis apparatus [2]. Table 1 lists the chemical composition of the 430-SS in weight percent. The starting dimensions of the stainless steel foils and Al foils are 0.66 mm thick and 50 mm × 50 mm squares and 0.81 mm thick and 50 mm × 50 mm squares, respectively, and the total thickness of the samples were ~12.6 mm with 9 layers of SS foil and 8 layers of aluminum layers. Foils were cleaned with wool felt and then rinsed in methanol and rapidly dried in order to remove some of the initial oxide layer and surface contaminants before processing.

The foil stack was placed between two, nickel alloy platens on the crosshead of a screw-driven load frame. Each platen was heated by a set of 7 cartridge heaters, and the temperature and pressure of the platens were controlled via a computer-control program. A ceramic fiber blanket surrounded the synthesis apparatus to reduce heat loss. After loading the foils, the pressure is first increased to 3.8 MPa by load control at room temperature to ensure good contact between foils. For each temperature range employed for the following reactions, two different temperature windows were explored to ensure the nature of the reaction (solid, semi-solid, or liquid) was occurring over the two temperatures. Reacting samples were interrupted at 2-h increments, then the samples were quickly removed from the platen press and quenched in water to halt the reactions. Therefore, for a given reaction type, two different temperatures were examined and three samples at each temperature were processed for different times (2 h, 4 h, and 6 h).

For the solid–solid reaction, the specimens were annealed at 645 °C for 6 h followed by 650°C for 6 h. The pressure is maintained at 3.8 MPa during the heating. The semi-solid reaction samples were heated starting at 655 °C corresponding to the eutectic temperature in the Al–Fe phase diagram, where a two-phase (eutectic) region is formed consisting of liquid aluminum and the $Al_{13}Fe_4$ intermetallic (e.g. [18]). After the initial pressure of 3.8 MPa is applied to the sample, the platen position is fixed, and therefore at the onset of the semi-solid reaction, the measured platen pressure begins to drop, which indicates the formation of liquid phases. The semi-solid reaction is sustained at 655 °C for 6 h and then increased to 660 °C and held for another 6 h. The interruption interval during the semi-solid reaction processing was maintained at 2 h.

The liquid reaction samples were initially heated at 600 °C for 1 h under a pressure of 3.8 MPa to allow the diffusion bounding of the layers and minimize internal oxidation between the layers. The reaction temperature was then increased to 665 °C for 6 h and

Table 1
Chemical composition of 430-stainless steel.

Element	Cr	Mo	Si	Mn	C	P and S	Fe
Content (wt%)	17.5	0.1	0.5	0.5	0.12	< 0.07	Balance