



Nanoscale structures of the interfacial reaction layers between molten aluminium and solid steel based on thermophysical simulations



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ARTICLE INFO

Article history:

Received 3 December 2016

Received in revised form

15 December 2017

Accepted 19 December 2017

Available online 21 December 2017

Keywords:

Thermophysical simulation

Interfacial reaction

Order–disorder transformation

Twinning

ABSTRACT

A novel thermophysical simulation method to precisely control the reaction conditions was designed to investigate molten aluminium/solid steel interfacial reactions. The reaction layers between the materials were investigated by high-resolution transmission electron microscopy assisted by advanced focused ion beam technology. For the first time, fibrous-ordered Fe₂Al₅ and Fe₄Al₁₃ intermetallics induced by an order–disorder transformation appeared in the disordered substrates of Fe₂Al₅ and Fe₄Al₁₃, respectively. High-density planar defects composed of stacking faults and twinings were found inside the Fe₄Al₁₃ grains. The (001) twinning as a type of growing twin was considered to be the sole operating mechanism during the solid/liquid interfacial reaction.

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1. Introduction

The composite structures formed by aluminium and steel provide substantial advantages in terms of density, cost and corrosion resistance, among others. As a main manufacturing technology of these structures, welding faces difficulties due to the formation of brittle intermetallic compound (IMC) layers during the interfacial reaction between molten aluminium and solid steel. The IMC layers severely reduce the mechanical properties of the joint. Therefore, the microstructures of the interfacial reaction layers, the interfacial reaction mechanisms, and IMC regulation are extensively investigated to suppress IMC growth or improve the properties of the IMCs.

Since the 1950s, the phase compositions [1–3], growth kinetics and mechanism [4–10], and dissolution behaviour [11,12] of the interfacial reaction layer were revealed by traditional methods, such as heat dipping and brazing. The η phase (Fe₂Al₅) adjacent to steel is confirmed as the dominant component of the interfacial reaction layers because of both its fast growth according to parabolic kinetics and finger-like morphology caused by accelerated diffusion along the [001] direction. Moreover, another thin layer near Al, which is frequently ignored in previous investigations, consists of a θ phase (Fe₄Al₁₃, which is sometimes referred to as

FeAl₃).

Several new discoveries in the field of interfacial reactions between molten aluminium and solid steel were reported using advanced analysis methods. Springer et al. investigated the formation and growth behaviour of the intermetallic phases during interdiffusion between low-carbon steel and aluminium alloys using electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM) using a focused ion beam (FIB) [13]. The authors found that the total width of the reaction layer was determined mainly by parabolic diffusion-controlled growth of the η phase, which exhibited orientation-dependent growth kinetics [13,14]. In addition, the group that used atomic probe tomography suggested that the aggregation of Si atoms at the grain boundaries of brittle intermetallic phases, especially at the θ/η interface, can play an important role in the suppression of the extremely rapid growth kinetics of the η phase [15]. Pasche et al. observed the formation of IMCs between solid iron and liquid aluminium by time-resolved in situ X-ray tomography combined with scanning electron microscopy (SEM) [16]. This group found that growth occurred towards the liquid and towards the solid after nucleation on the liquid side at the interface occurred. On the metal solid side, growth began with a specific tongue-like feature that progressively thickened. This thickening was linked to the deformation of the iron matrix during the formation of the intermetallic compound. Growth in the direction of the liquid was slowed by erosion. In our previous study [17], we have found that an intensification of the dissolution led to the transformation of Fe₄Al₁₃/liquid aluminium

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into Fe_2Al_5 /liquid aluminium in the solid/liquid structure following an increase in the reaction temperature. The formation of $\text{Fe}_4\text{Al}_{13}$ adhering to the interface is governed by both reaction diffusion and precipitation behaviour at low temperatures. However, the results indicated that precipitation behaviour became the sole formation mechanism of $\text{Fe}_4\text{Al}_{13}$ at high temperatures.

All the abovementioned studies with relatively long times (a few minutes) and low temperature reactions were investigated using traditional investigation methods, such as heat dipping and furnace brazing. In recent years, several technologies with short reaction times, high reaction temperatures (near or above 1273 K), and high heating/cooling rates (several hundred K/s), such as laser/arc brazing and bimetallic manufacturing and coating, have been developed to effectively suppress the growth of brittle IMCs and thus have been attracting significant attention. To gain insight into the mechanisms, kinetics, and imbalance of the solid/liquid interfacial reaction of these technologies, traditional investigation methods are insufficient. Moreover, previous investigations regarding interfacial reactions between molten aluminium and solid iron have been performed only at the microscale, and attention to fine structures (e.g., nanoscale) was limited. Thus, investigations regarding nanoscale structures can contribute to the in-depth analyses of the interfacial reaction mechanism and prompt the development of new technologies to control such reactions.

In the present study, a novel method with high-precision control of the reaction conditions was introduced to investigate the molten aluminium/solid-steel interfacial reaction during a short duration. The structures of interfacial reaction layers between molten aluminium and solid steel were investigated by high-resolution transmission electron microscopy (HRTEM). The nanoscale structures and formation mechanisms of interfacial reaction layers were subsequently analysed.

2. Experimental details

Low-carbon steel bars with 10 mm diameters were selected as the experimental samples. The low-carbon steel was composed of C (0.14–0.22), Mn (0.30–0.65), Si (≤ 0.30), S (≤ 0.050), and P (≤ 0.045) and balanced with Fe (wt.%). A keyway was machined in this bar, as shown in Fig. 1a. A pure aluminium (99.99 wt%) block was processed to fit the keyway size by line cutting. An ethyl alcohol-mixed NOCOLOK flux was painted on the surface of the keyway and dried. After embedding the aluminium block in the pinch of the keyway, the keyway was closed by a steel sheet with the same chemical composition.

Subsequently, a renovated thermal simulator, Gleeble 1500, was used to heat the steel bar, as shown in Fig. 1b. A resistance heating mode on the specimen was used, and the highest heating rate reached was 10000 K/s. The controlling thermocouple was welded in the steel near edge of the keyway to reduce the measuring position errors. A water-spray nozzle that automatically controlled by the system was added to regulate the cooling rate of the specimen. Compared with conventional methods, this method can precisely control the reaction temperature and reaction time at high heating/cooling rates. Fig. 1c compares the controlled and measured curves of the temperature. Our method showed high-precision control for the solid/liquid interfacial reaction and was thus highly suitable to investigate the non-equilibrium interfacial reaction and reaction kinetics during short durations (a few seconds).

The specimens were systematically sectioned by a line cutting machine and were mounted, grinded, and polished using conventional techniques. The microstructures were examined using an SEM system attached with an energy-dispersive X-ray spectrometer. The nanoscale-sized structures were analysed by TEM bright

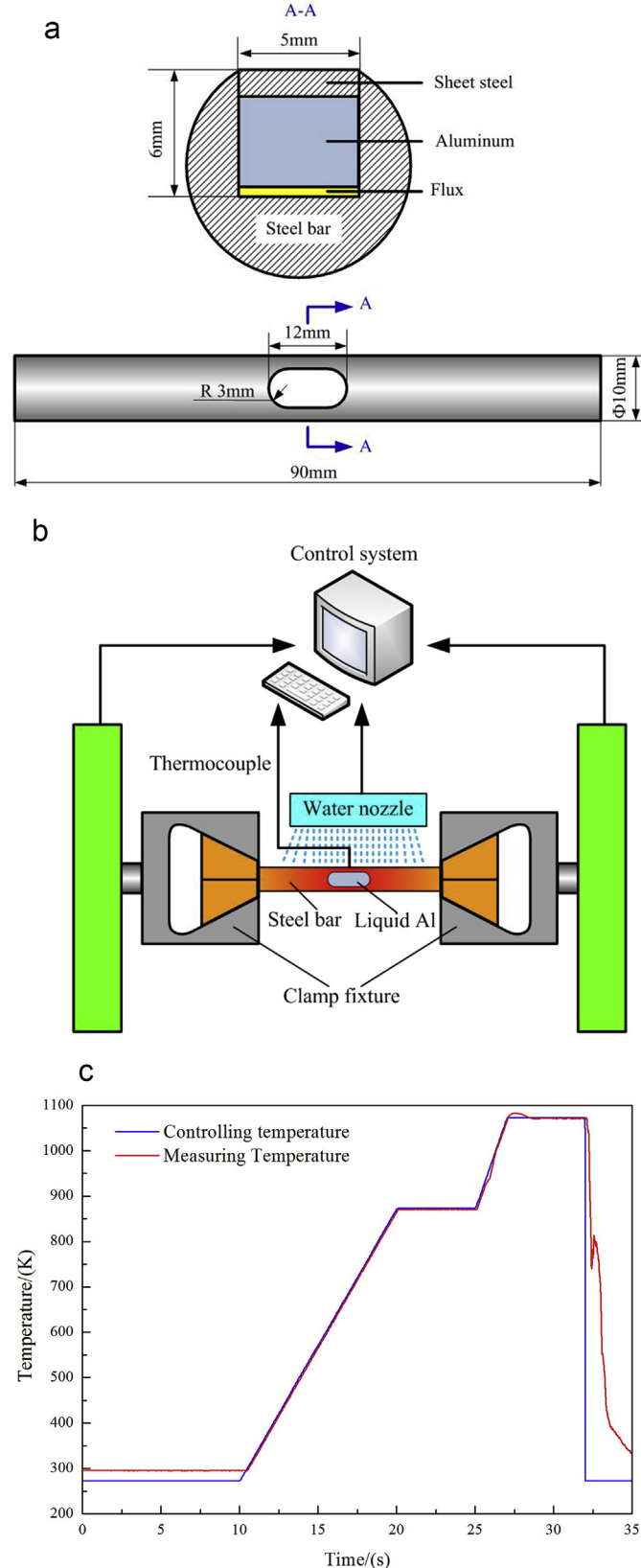


Fig. 1. Schematic of the interfacial reaction between solid iron and molten aluminium: (a) specimen, (b) heating principle, and (c) comparison between the controlling and measuring temperature curves.

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