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Amorphous iron nanoparticles formed on aluminum surfaces during thermomechanical treatment

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ABSTRACT

Iron nanoparticles were observed on the surface of commercial purity aluminum alloys after thermomechanical treatment using a hot rolling tribo-simulator with a roll surface roughness (R_a) of 0.01 μm. These nanoparticles were spherical in shape and possessed an amorphous structure. They were observed lying on the oxide layer of the alloy surface and on crack faces. Iron nanoparticles have not been previously reported on aluminum surfaces under this thermomechanical condition. The surface of the hot rolled aluminum alloy was also covered with fractured iron-rich intermetallic particles embedded into the surface and caught within cracks.

1. Introduction

The tribological interactions that occur during rolling and subsequent metal forming operations are influenced by the near-surface oxides developed on the alloy surface prior to and during rolling [1–3]. Though temperature has been noted to be the major stimulus for oxide growth on aluminum alloy surfaces, alloying additions have been observed to influence oxide nucleation, oxidation rates and composition [4,5]. The levels of these alloying additions at the alloy surface are also influenced by the thermomechanical treatments and the diffusion coefficients of these elements in aluminum at elevated temperatures [6]. Intermetallic particles have also been reported to alter the local properties of the oxide layers [7]. The composition of the oxide layers is also affected by the absorption of components from lubricants i.e., the formation of chemisorbed species on the rolled aluminum surface [2,8,9]. It is, therefore, vital to characterize the surface/near-surface features developed during the tribological contact of thermomechanical processes.

This work presents the characterization of iron nanoparticles observed on the surface of a commercial purity aluminum alloy after a typical thermomechanical process. The presence of these iron nanoparticles on rolled aluminum surfaces has not been previously reported. The synthesis of these metal nanoparticles has been considered a challenge due to the extremely rapid cooling and small nucleation rates required [10].

2. Experimental procedure

A rolling tribo-simulator with a roll-on-block configuration previously described in detail by Riahi et al. [11] was used to carry out the rolling tests. The roll material was machined from AISI 52100 steel to a diameter of 21 mm. A commercial purity aluminum alloy (AA1100), machined into blocks 10 mm wide, 30 mm thick and 95 mm long, was used as the work piece. The surface of the work roll was polished to a roughness (R_a) of 0.01 μm to eliminate the effect of roll roughness. The work roll was cleaned with a 15% (wt/wt) sodium hydroxide solution after each pass to remove aluminum transfer and then polished to maintain the surface roughness. The aluminum specimens were polished to a surface roughness (R_a) of 0.02 μm. The first pass was performed at 550 °C with a 10 °C temperature reduction at each subsequent pass such that the temperature at the final (tenth) pass was 460 °C. Lubrication was provided by an oil-in-water emulsion with a 4% (v/v) concentration, and a forward slip of 7% was used in each pass. The specimen surfaces were examined with an FEI Quanta 200 FEG environmental scanning electron microscope (SEM). The polished aluminum surface (Fig. 1) was covered with intermetallic particles, which were rich in iron. Samples were then prepared by the lift-out method, in which the surface was protected by depositing a thin layer of carbon on the area of interest and cross-

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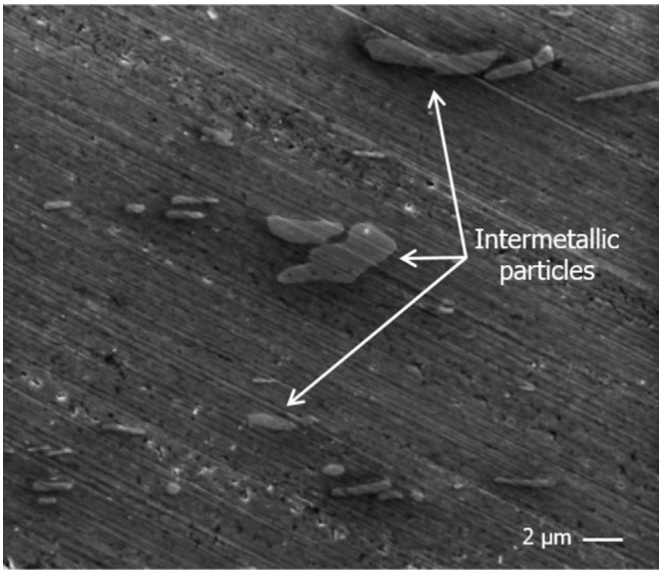

Fig. 1. Scanning electron image of the polished untreated AA1100 alloy surface displaying the intermetallic particles on the surface.

sectional trenches were ion milled using the FIB H-bar method. The samples were then examined using an FEI Titan 80-300LB Transmission Electron Microscope (TEM).

3. Results and discussion

Surface examination of the rolled AA1100 sample under a scanning electron microscope (SEM) revealed the rolled surface was covered with microcracks occurring transverse to the rolling direction (Fig. 2(a)). These microcracks have been attributed to grain boundary sliding (GBS), inducing steps between and within grains, during the first rolling pass [12]. Fractured intermetallic particles were observed randomly dispersed across the alloy surface (Fig. 2(a)). A closer examination (Fig. 2(b)) revealed debris from the fractured intermetallic particles embedded into the alloy surface and within microcracks. Intermetallic debris particles were also observed within nanocracks covering the surface of the alloy at this magnification (Fig. 2(b)). The high shear stresses experienced during thermomechanical processing are known to cause the continuous fracture of intermetallic particles over the course of the processing schedule. Fig. 2(c) displays several fractured intermetallic particles and debris embedded in the sample surface at

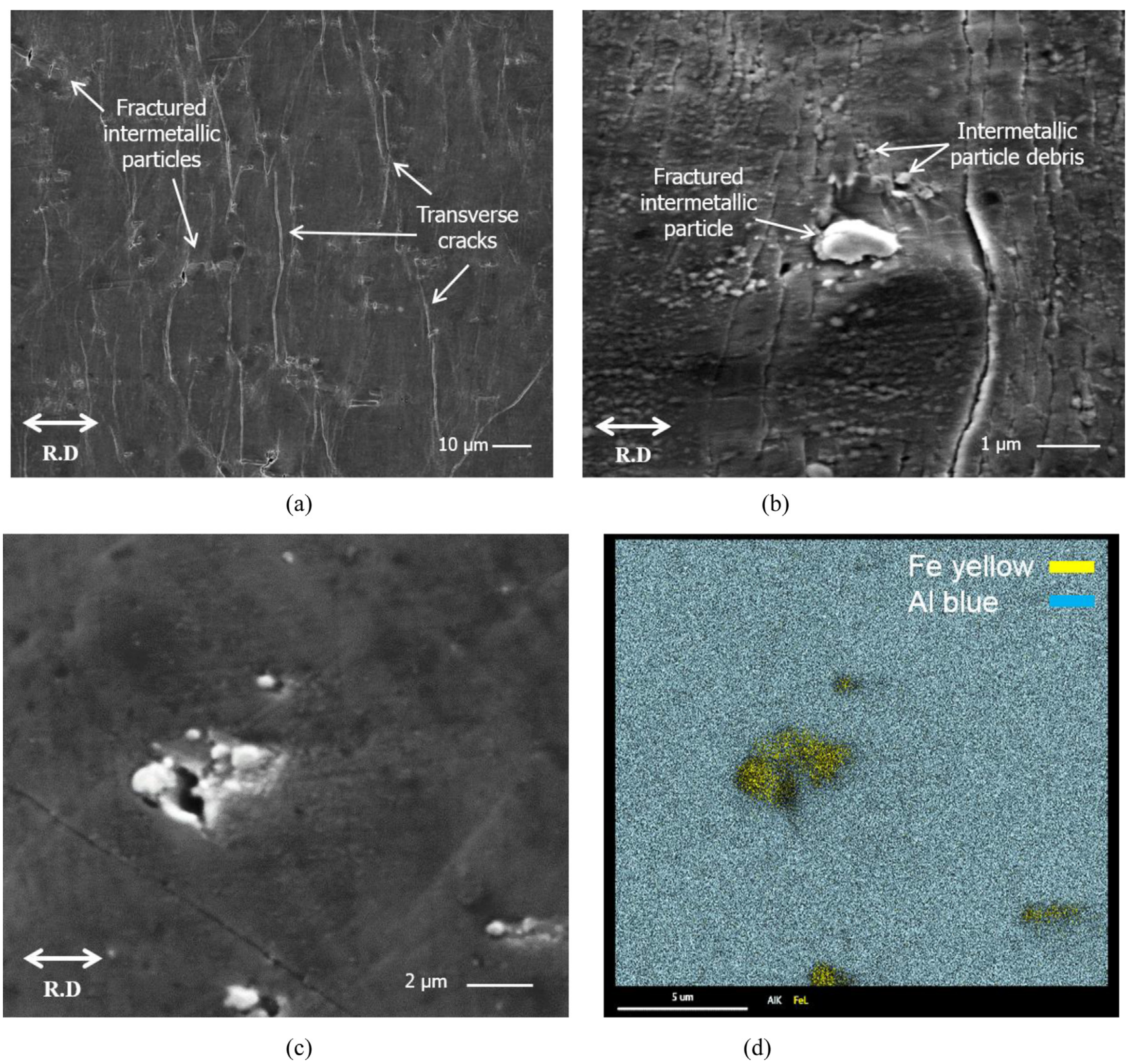

Fig. 2. Scanning electron images of the AA1100 alloy surface after a 10 pass hot rolling schedule, showing (a) surface covered with transverse cracks, (b) fractured intermetallic particles and debris, (c) higher magnification of intermetallic particle fractures embedded in the surface and (d) EDS map of area shown in (c) displaying iron content of intermetallic particles.