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Abstract

The coating layer of Fe–Al powders on the steel substrate was prepared by mechanical alloying at room temperature. Fe, Al, and the steel substrates were milled with high-energy ball milling for 32 h with a ball-to-powder ratio of 8 in an argon atmosphere to prevent oxidation during milling. Although mechanical alloying was performed for 32 h, no new phases were observed after mechanical alloying, as analyzed by X-ray diffraction. However, the crystallite size of the milled powders for 32 h decreased by factor two compared with the initial powders. Scanning electron micrographs showed that the coating layers formed >8 h after mechanical alloying. The intermetallic Fe_3Al formed after the substrate was annealed at 500 °C.

Abstrak

Pelapisan Serbuk Fe–Al yang Dibantu dengan Pemaduan Mekanik pada Substrat Baja. Lapisan yang telah dilapisi serbuk Fe–Al pada substrat baja dipreparasikan menggunakan metode paduan mekanik pada suhu ruang. Fe, Al dan substrat baja digiling menggunakan *high-energy ball milling* selama 32 jam dengan rasio antara bola pada serbuk 8 dan atmosfer argon untuk mencegah oksidasi selama proses penggilingan. Berdasarkan analisis difraksi sinar X, tidak ada fasa baru yang terbentuk setelah proses pemaduan mekanik selama 32 jam. Namun ukuran kristal serbuk yang digiling selama 32 jam menurun dibandingkan serbuk awal. Citra *scanning electron microscope* menunjukkan bahwa lapisan dapat terbentuk setelah pemaduan mekanik lebih dari 8 jam. Fasa intermetalik Fe_3Al terbentuk setelah proses anil pada suhu 500 ºC.

Keywords: coating, steel substrate, mechanical alloying, Fe–Al powder, intermetallic

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1. Introduction

Fe–Al alloys are structural materials with excellent properties, such as resistance to oxidation, sulfidation, and corrosion, for high-temperature applications [1,2]. Given these advantages, Fe–Al alloys are considered to substitute Ni-based superalloys. The fabrication of Fe–Al alloys requires advanced melting techniques, such as air-induction melting, vacuum-induction melting, vacuum arc melting, vacuum arc double melting, and Exo-Melt™ process [1], [3]–[7]. However, the melting point difference between Al and Fe is another aspect that needs to be considered for the melting technique. For instance, Al and Fe have a melting point of 660 °C and 1536 °C, respectively. Therefore, a special treatment is needed to fabricate FeAl alloys via the casting method. Pure Al ingot was melted in the furnace at 730 °C in Ar atmosphere [8], and then Al-10Fe master alloy was added and mixed with the ceramic rod. Continuous rheo-extrusion was used to form FeAl alloy rod [8], [9], in addition to pouring the molten cast to the mold.

Mechanical alloying is a well-known technique used to synthesize alloys from the powder route [10] through high-energy ball milling. Solid-state reaction occurs during high-energy ball milling because of the impact of energy and ball friction. Mechanical alloying is suitable for two powders with a large difference in melting point. Therefore, powder metallurgy is an alternative method for the fabrication of Fe–Al alloys [11]–[18]. Ball milling also gives a coating effect on ceramics and metals [10], [19]. This technique is expected to form a coating layer at room temperature. Several studies reported about coating-assisted ball milling. Canacki *et al*. reported the formation of intermetallic coating layer compounds based on Fe and Al after ball milling for 20 h (pre-milling 16 h and coating 4 h) [17]. Zhan *et al*. used a vibrator mill equipped with a heater for the fabrication of Fe–Al intermetallic coating [20]. Compared with pack cementation, Fe–Al intermetallic coating can be synthesized at low temperatures (440–600 ℃) in a short time (15–120 min) [20]. Aryanto *et al*. [21] found that Fe–Al coating in the steel substrate improves the oxidation properties significantly. The weight gain after oxidation can be decreased by a factor of 10 for the substrate coated with MA relative to the substrate without coating [21]. The present study aims to investigate the possibility of coating Fe–Al powder and the formation of intermetallic on structural steel prepared by mechanical alloying. Mechanical alloying was performed by high-energy ball milling (HEM), which was developed by the authors [22]. This technique is unique due to 3D ellipse motion, which enhances mechanical alloying. Data on the powder evolution, phase, coating thickness, and hardness are presented.

2. Experimental

Commercial structural steels (AISI 1045) were used in this study as a substrate. The substrate diameter and thickness were 10 and 3 mm, respectively. Prior to mechanical alloying, the substrate was mirror-polished using sandpaper until #1000. The coating powder, Fe powder (>99% purity, Merck, Germany) with average particle size 10 µm and Al powder (>90% purity, Merck, Germany), was mixed with a ratio of 60:40 in atomic weight. HEM (HEM-E3D; Research Center for Physics; Indonesia) was used for mechanical alloying. The ball-to-powder ratio was 8 with a powder amount of 15 g. The ball was 5 mm in size and made of stainless steel and the jar. The powder and substrate were milled at a speed of 263 rpm in an argon atmosphere to prevent oxidation.

The powder and substrate were analyzed at 4, 8, 16, and 32 h. High-energy milling was automatically turned off for 1 min every 5 min of alloying to minimize increasing temperature during milling. The mechanically alloyed substrate at 32 h was annealed at 500 ℃ for 2 h in an argon atmosphere.

The crystalline phases of the powder before and after mechanical alloying were identified by X-ray diffraction (XRD; XD610; Shimadzu; Tokyo; Japan) with a CuKα radiation source. The microstructures of the powder and substrates were observed using scanning electron microscopy (SEM; JSM-6390A; Jeol; Tokyo; Japan). An energy-dispersive X-ray (EDX) analyzer installed in the SEM was used to determine the chemical compositions of the coating layer on the substrate. The hardness of the coating layer before and after annealing was measured by Vickers hardness tester (MXT50; Matsuzawa; Japan).

3. Results and Discussion

Figure 1 shows the XRD patterns of Fe–Al powder with various mechanical alloying times. Indeed, the peaks of Al (ICDD # 01-089-2837) broadened with prolonged mechanical alloying time, indicating that the crystallinity of Al powder reduced due to heavy milling, as shown in Figure 1. By contrast, the peaks of Fe powder (ICDD # 01-085-1410) did not broaden as much as those of Al powder probably because of the softer characteristic of Al powder than Fe powder. The factors influencing peak broadening are crystallite size, instrument error, and microstrain. Crystallite size and microstrain can be estimated using Equations (1) and (2):

$$\mathrm{B} = \frac{0.94 \times \lambda}{\mathrm{D} \cos \theta} \tag{1}$$

$$\mathrm{B} = 4\varepsilon \tan \theta \tag{2}$$

where B is the full width at half maximum (rad), λ is the wavelength of X-ray source (nm), D is the crystallite size (nm), and θ is the peak angle. Combining those equations, we obtained Equation (3):

$$\mathrm{B} \cos \theta = \frac{0.94 \times \lambda}{\mathrm{D}} + 4\varepsilon \sin \theta \tag{3}$$

According to Williamson-Hall method, Equation (3) is a linear equation where the y-axis is B cos θ, and the x-axis is sin θ. Therefore, the slope is microstrain, and the intercept is crystallite size. The crystallite sizes of milled powders for 4, 8, 16, and 32 h were 37.6, 30.3, 23, and 16.6 nm, respectively, showing that the crystallite size decreased by factor two after mechanical alloying for 32 h.

Figure 2 displays the evolution of the powder during mechanical alloying. As shown in Figure 2 (a), the powder was spherical before mechanical alloying. However, after mechanical alloying for 4 h, it became flat, as shown in Figure 2 (b). The change in powder shape was due to the impact energy of the ball during mechanical alloying. The flat shape increased with increasing milling time, which indicates the initial process of mechanical alloying. Further milling time, for instance, 8 h (Figure 2 (c)) and 16 h (Figure 2 (d)), agglomerated powder observed as a consequence of fracture and cold welding during mechanical alloying. The final particle size of the milled Fe–Al powder is shown in Figure 2 (e).

The powder was highly agglomerate with a particle size of more than 5 µm, which is larger than the starting powder. According to Suryanarayana [10], mechanical alloying has four stages. First, the powder became flat due to the micro-forging during the early stage of mechanical alloying. In the second stage, the particle size increased due to the formation of composite lamellar structure from the flattened powders during cold welding. The next stage is the hardening of the lamellar structure. Thus, the hardness of the powder increased. The last stage is the convolution of the lamellar structure due to excessive welding without preference orientation.

Alloying started at this stage, along with the saturation of hardness and particle size. Hence, we can conclude the mechanical alloying of Fe–Al in this study occurred until the second stage, as shown in Figure 2 (e). This result is in agreement with the phase analysis by XRD, where no new phase formed during the mechanical alloying of Fe–Al powder. Therefore, Fe–Al intermetallic possibly formed after > 32 h of high-energy milling.

Figure 3 displays the SEM images of the substrate surface after mechanical alloying for 4 h (Figure 3 (a)) and 32 h (Figure 3 (b)). Indeed, the presence of powder on the substrate surface increased with prolonged milling time, as shown in Figure 3. The surface of substrate cover by the powder after 32 h of mechanical alloying is shown in Figure 3 (b). The thickness of coating layers is shown in Figure 4. As expected from the ball milling results, no coating layer was observed at 4 h (Figure 4 (a)) and 8 h (Figure 4 (c)) of mechanical alloying. The coating layers appeared after > 8 h of mechanical alloying, as shown in Figure 4 (c) and (d). The initial mechanical alloying was indicated by the small coating layer onto the ball. Therefore, the first stage of mechanical alloying started at > 8 h, as shown in Figure 4 (c). The thickness of the coating layers at 4 and 8 h were 20.5 ± 4.2 and 51.5 ± 14.1 µm, respectively.

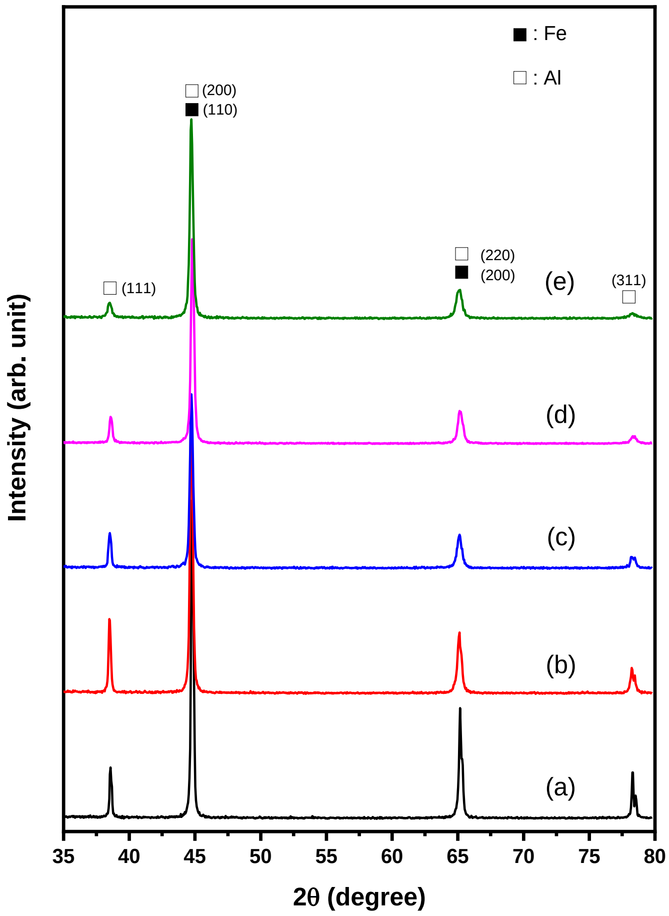

Figure 1. XRD Patterns of Fe–Al Powder at Milling Time (a) 0 h, (b) 4 h, (c) 8 h, (d) 16 h and (e) 32 h

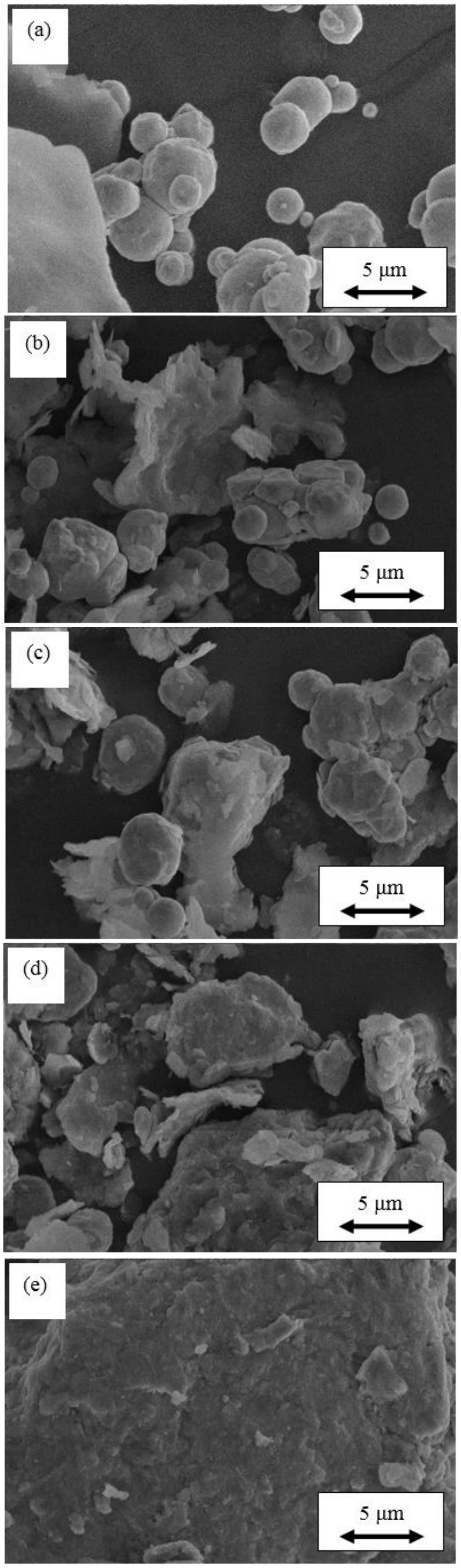

Figure 2. SEM Images of Fe–Al Powder Before (a) and After Milling for (b) 4 h, (c) 8 h, (d) 16 h, and (e) 32 h

Fe–Al intermetallic was not formed during mechanical alloying. The substrate was annealed at 500 °C for 2 h in Ar atmosphere. Figure 5 shows the SEM image of the annealed substrate. As shown in Figure 5, three colors of phases were observed: white (001), gray (002), and black (003). EDX analysis showed that the white, gray, and black corresponded to Fe, $Fe_{1.5}Al$, and Fe_3Al, respectively. The hardness of the coating layer improved after annealing. The hardness values of coating layer before annealing in the outside layer and near interface were 50.1 ± 1.7 and 41.5 ± 3.1 VHN, respectively, and 71.5 ± 1.8 and 61.6 ± 0.7 VHN after annealing, respectively. The increase in hardness of the coating layer suggests the formation of a new phase because the intermetallic phase is harder than elemental Fe and Al (Table 1).

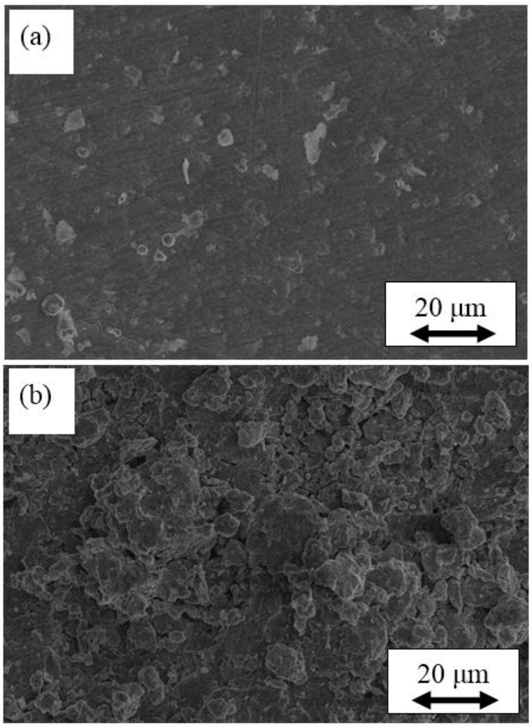

Figure 3. SEM Images of Steel Surface After Milling (a) 4 h and (b) 32 h, Showing the Surface Covered by the Powder After Milling for 32 h

Table 1. EDX Analysis of Annealed Substrates

Point	Element (wt. %)			
	Fe	Al	O	Total
001	100	-	-	100
002	66.32	20.30	13.38	100
003	31.93	46.18	21.89	100

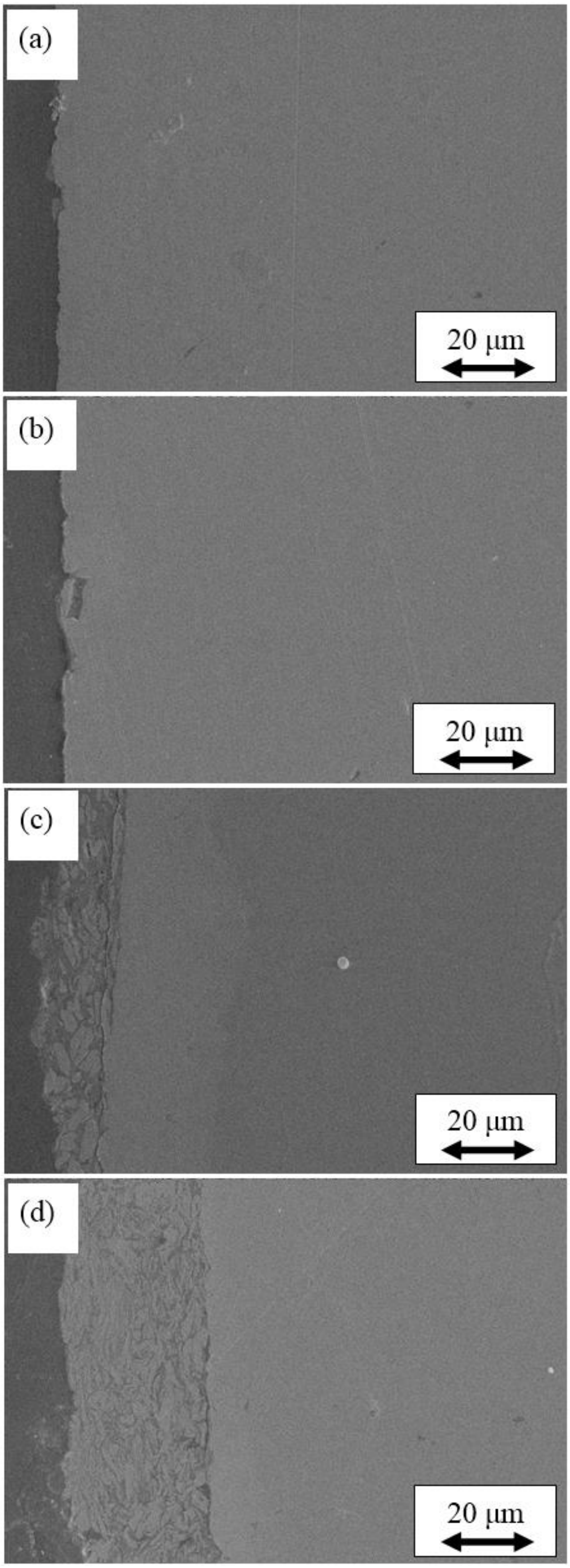

Figure 4. SEM Images of Cross Section of Steel After Milling (a) 4 h, (b) 8 h, (c) 16 h, and (d) 32 h

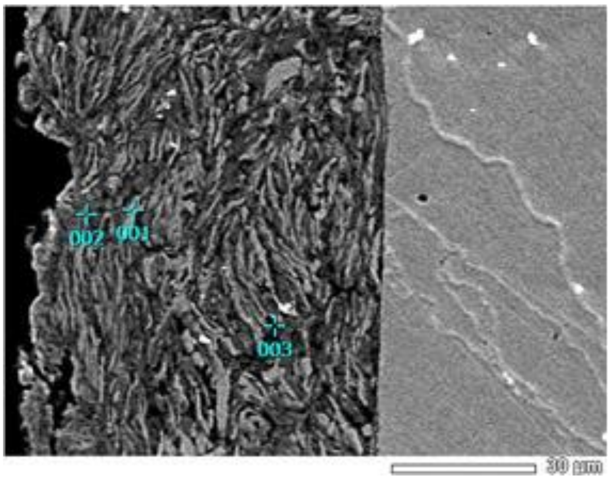

Figure 5. SEM Images of Cross Section of Annealed Substrate Steel

4. Conclusion

Steel substrate was successfully coated by Fe–Al powder through mechanical alloying. The coating layer started to form > 8 h after mechanical alloying. The thickness of the coating layer at 16 and 32 h of the alloying time were 20.5 ± 4.2 and 51.5 ± 14.1 µm, respectively. However, the intermetallic phase did not form after alloying for 32 h, and only the broadened XRD peaks were observed. This phenomenon is because the mechanical alloying for 32 h only reached the second stage of mechanical alloying, which showed the agglomerated powder with a particle size larger than the starting powder. Therefore, the alloying time must be extended to allow the formation of intermetallic Fe–Al. In addition, Fe_3Al intermetallic formed after annealing of a substrate containing Fe–Al coating layer at 500 ℃ for 2 h in Ar atmosphere.

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