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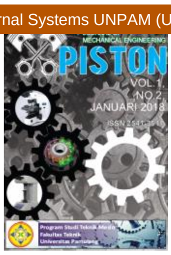

Correlations between Structural and Hardness of Fe-50%Al Coating Prepared by Mechanical Alloying

Didik Aryanto[1,2,*], Toto Sudiro[1], and Agus Sukarto Wismogroho[1]

[1]*Research Center for Physics, Indonesian Institute of Sciences, Serpong 15314,*
Tangerang Selatan, Banten, Indonesia
[2]*Mechanical Engineering, Faculty of Engineering, Universitas Pamulang*
Jl. Surya Kencana No. 1 Pamulang, Tangerang Selatan, Banten, Indonesia

E-mail: *didik_phys@yahoo.co.id

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Abstract: Fe-50%Al coatings were deposited on the surface of low-carbon steel using mechanical alloying technique at different milling times. The correlation between structure and hardness of coating before and after heat treatment was investigated. At the milling time of less than 180 min, the coating has an elongated lamellar structure. The size of elongated lamellar structure decreased with increasing milling times which led to an increase in the hardness value of coating. After heat treatment, the coating transformed to FeAl intermetallic phase with a denser structure and uniform in the composition. It affected the hardness of coating. The hardness value of all samples after heat treatment was higher than coating after milling. The hardness of coating was strongly influenced by the morphology and phase of coating.

Keywords: Fe-50%Al, coating, structure, hardness, mechanical alloying

Abstrak: Coating *Fe-50%Al telah dideposisi pada permukaan* low-carbon steel *menggunakan metode* mechanical alloying *dengan variasi waktu* milling*. Hubungan antara struktur dan kekerasan dari* coating *sebelum dan setelah perlakuan panas telah diteliti. Pada kondisi waktu* milling *kurang dari 180 menit,* coating *tersebut memiliki struktur lamellar yang memanjang. Ukuran struktur lamellar yang memanjang tersebut menurun dengan meningkatnya waktu* milling, *yang mempengaruhi kenaikan tingkat kekerasan dari* coating*. Setelah proses perlakuan panas,* coating *tersebut bertransformasi menjadi fasa i FeAl* ntermetalic *dengan struktur yang lebih padat dan komposisi yang seragam. Hal ini mempengaruhi kekerasan dari* coating*. Nilai kekerasan dari seluruh sampel setelah perlakuan panas lebih tinggi dibandingkan* coating *setelah milling. Kekerasan dari* coating *sangat dipengaruhi oleh morfologi dan fasa* coating *tersebut.*

Kata kunci: *Fe-50%Al,* coating, *struktur, kekerasan,* mechanical alloying

INTRODUCTION

Low carbon steel is extensively utilized in the construction sectors and various applications, due to their low cost than high-alloy steels and good mechanical properties as well [1,2]. However, low carbon steel has poor oxidation and corrosion resistance especially at high temperature, which restricts to their applications. Investigation on the fabrication of high resistant coating against oxidation and corrosion on low carbon steel is considered as an essential and promising task for solving the problem. Metals which can form continuous and stable oxides are widely believed to be suitable for fabricating anti-oxidation and corrosion coatings [3]. Aluminium (Al)-based can be apply as coating to isolate and act as a shield against the aggressive corrosive elements. This is due to their ability to form highly densified protective α-Al_2O_3 layers. Al-coatings offer a combination of attractive properties such as low density, ease of fabrication and resistance to oxidation at high temperature [4], but their have poor mechanical and physical properties. One way to improve the mechanical and physical properties of Al coating at high temperatures is carried out by adding Fe element. The presence of Fe in Al matrix results in the formation of secondary phases, which causes the microstructural stability of Al-Fe [5]. In addition, the previous study reported that the grain refinement to the sub-micrometer or even to the nanocrystalline size can improve the mechanical and physical properties of Al-alloys [6]. Mechanical alloying is one of the top-down techniques used for reducing the grain size of the materials. This method can be used to produce ultra fine and uniform powders, which is now widely applied for preparation of intermetallics, extended solid solutions and amorphous phases of powders [3,7].

Recently, mechanical alloying method has been used to deposit thick multi-component structures with high bond ability on different substrates, such as TiAl on Ti substrate [8], TiCr and TiCu on Ti-6Al-4V substrate [9], NiAl on carbon steel [10,11], Al on low carbon steel [1], Cr on Cu substrate [12], hydroxyapatite on Ti substrate [13], CrAl on low carbon steel [2], CrAl on Cu substrate [3], and TiC on AISI D2 steel substrate [14]. The present work focuses to discuss the correlation between structural of Fe-50%Al coatings before and after annealing at 600°C and their hardness.

EXPERIMENT

A detail of experimental procedure for the sample preparation has been described elsewhere [15]. Here, we briefly repeated the experimental. For the experiment, low-carbon steel plates were used as substrate with a dimension of 10×8×3 mm^3. Before coating process, the surface of the steel plates was polished using SiC papers for up to No. 1200 and cleaned with standard cleaning method to remove contaminants from the surface. The commercial Fe (99,5% purity) and Al (99,5% purity) powders were used as raw material. In order to deposit Fe-Al coating on the surface of low carbon steel, the two steps of milling were carried out using high speed shaker mill.

The phase structure of Fe-50%Al coating before and after heat treatment was identified by X-ray diffraction (XRD) (SmartLab-Rigaku Co. Ltd., Japan), using a continuous scan mode. The morphology of the Fe-50%Al coatings was characterized from cross-sectional sample using scanning electron microscope (SEM-Hithaci High-Tech Co. Ltd, Japan) with back-scattered electron (BSE) mode. The hardness of Fe-50%Al coating was measured from the cross-sectional sample using a Vickers Microhardness tester (LM-100AT) at a load of 500 kgf and a dwell time of 15 s.

RESULT AND DISCUSSION

Figure 1 shows the XRD pattern of low carbon steel plate. The 2θ values were compared to the standard ICDD data card (04-014-0360). The XRD pattern of low carbon steel plate exhibits sharp and narrow peaks. They have a strong preferred orientation along the (110) plane at 2θ = 44.67°. Other diffraction peaks, (200) and (211), with less intensity were also found. The results indicate that the low carbon steel exhibits a polycrystalline with a body-centered-cubic (bcc) structure.

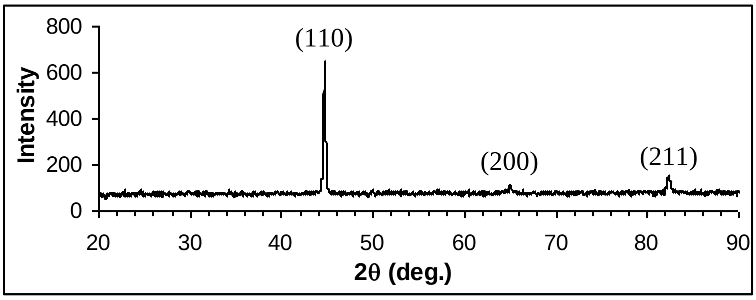

Figure 1. XRD pattern of low carbon steel plate.

Figure 2 shows the morphology from cross-section of Fe-50%Al coating prepared at different milling times. The coating thickness and formed phase on Fe-50%Al coating after mechanical alloying were discussed elsewhere [15]. The summarizing results are given in Table 1. The XRD analysis (in the Table 1) shows the diffraction peak of Al dan Fe. This provides an evidence of the deposition of Fe-Al coating on the low carbon steel surface. The presence of Al phase implied that no intermetallic phase formed during mechanical alloying process [3]. The Fe and Al element in the Fe-50%Al coating is separated when the milling time is less than 180 min. Liu et al. show that an elongated lamellar of the Fe-40Al-5Cr powder revealed that the element is not reacted and difference in the composition [16]. The cross-section Fe-50%Al coating in this research supports that the Fe-Al element is not reacted and difference in the composition. After milling for 180 min, the Fe(Al) solid solution is formed. This could be related to the dissolution of Al into Fe. The result also shows that the elongated lamellar structure becomes finer, resulting uniform microstructure (Figure 2d). The Fe-50%Al powder particles trapped between the ball and substrate prior to adhere on the substrate surface [1]. Increasing the duration of mechanical milling, the collision between ball and substrate leads to increase in the plastic deformation. It causes that the particle had a tendency to consolidate into bulk material and the coating layer was denser (Figure 2d) [10].

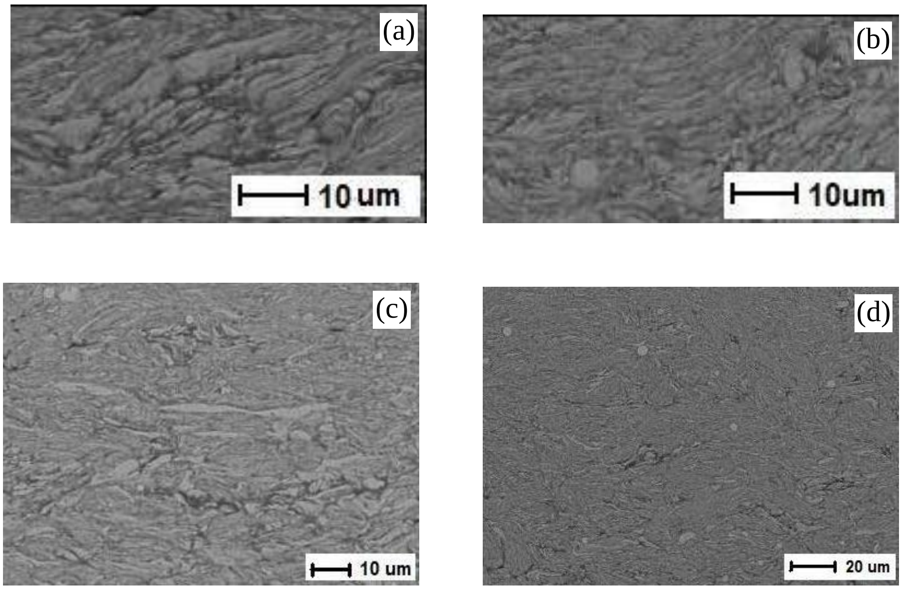

Figure 2. Microstructure features from cross-section of Fe-50%Al coating on low carbon steel prepared at different milling time for (a) 30 min, (b) 60 min, (c) 120 min, and (d) 180 min.

Table 1. Phase structure of Fe-50%Al coating prepared by MA at different milling times.

Process type	Thickness (μm)	Phases	Structure
MA for 30 min.	38.71	Fe, Al	bcc/bcc
MA for 60 min.	92.90	Fe, Al	bcc/bcc
MA for 120 min.	103.87	Fe, Al	bcc/bcc
MA for 180 min.	141.94	Fe(Al)	Bcc

The structure evolution of Fe-50%Al coating after heat treatment at 600ºC for 2 h was investigated [15] and the summarizing results are given in Table 2. From XRD analysis, the Fe and Al element transform into FeAl intermetallic phase after heat treatment. No phase related to Al, Fe, or oxidation product of Fe and Al were found in the XRD analysis of all samples. Similar result is shown by Haghighi et al., the formation of FeAl intermetallic was occured at the Fe-50 at.%Al powder after annealing process [17]. During heat treatment, a solubility of Al in Fe matrix affects the formation of secondary phase between Fe and Al [5]. Similar result was shown in the NiAl coating on Ti substrate by Mohammadnezhad et al. [10]. The dissolution of Al into Ni caused the formation of NiAl intermetallic. SEM image from cross-section of the Fe-50%Al coating at varying milling times after heat treatment at 600ºC for 2 h are shown in Figure 3. The elongated laminar structure is not observed in the microstructure of Fe-50%Al coating after heat treatment. However, some larger size granular and porosity were observed in the microstructure of all samples. The diffusion of Al in the Fe matrix is believed affect the formation of porosity. The larger granular may come from the original structure of Fe-50%Al coating before heat treatment. Compared with Fe-50%Al coating after milling, coating after heat treatment has uniform structure. It is shown by the uniform colour in the BSE SEM images. The results of SEM characterization confirm the results of XRD analysis, which shows that the Fe and Al element completely reacted to form FeAl intermetallic.

Table 2. Evolution of structure of Fe-50%Al coating prepared at different milling times after heat treatment at 600°C for 2 h.

Process type	Phase	Structure	Crystalline size (Å)	Lattice strain (%)
MA for 30 min.	FeAl	Bcc	5.16373	0.1787
MA for 60 min.	FeAl	Bcc	4.89214	0.1890
MA for 120 min.	FeAl	Bcc	3.44234	0.2682
MA for 180 min.	FeAl	Bcc	4.92384	0.1879

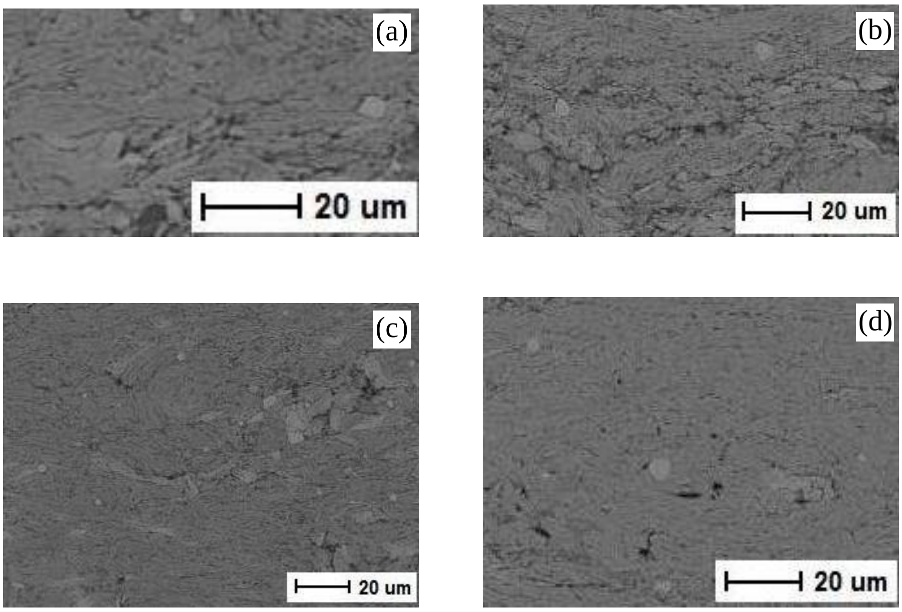

Figure 3. Microstructure features from cross-section of Fe-50%Al coating on low carbon steel prepared at different milling time for (a) 30 min, (b) 60 min, (c) 120 min, and (d) 180 min after heat treatment.

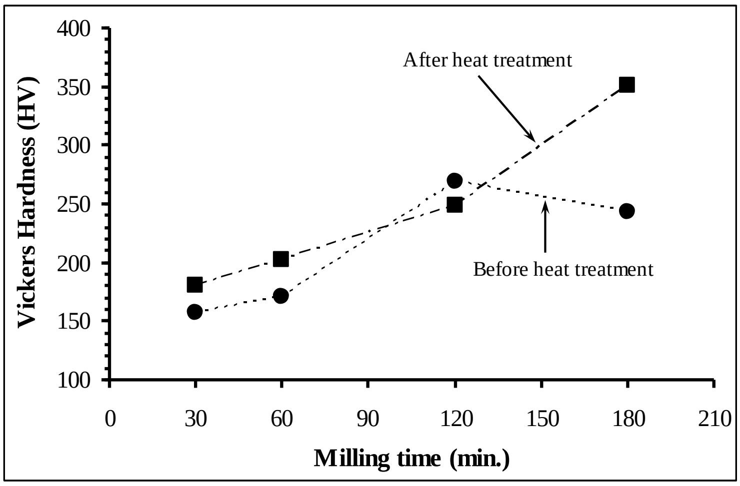

Figure 4. Vickers Hardness of Fe-50%Al coating on low carbon steel prepared at various milling time before and after heat treatment at 600°C.

Vickers hardness testing was carried out to evaluate the hardness of the Fe-50%Al coating. Figure 4 shows the result of cross-section microhardness of Fe-50%Al coating at different milling times as milled and after annealing. The hardness of Fe-50%Al coating after milling increases with increasing milling time. It may be attributed to the reduction in the grain size of coating (as show in the Figure 2a-d) [18]. After milling time for 180 min, however, the hardness of Fe-50%Al is decreased. Similar result was shown in the NiAl coating prepared using a mechanical alloying technique [10]. The optimum coating hardness is obtained at specific milling time, after that the coating hardness decreases. The different between the compressive residual stresses in the coating prepared at different milling time is believed to affect the hardness of the coating [10,12]. Other studies show that the smallest grain size leads to some softening for finest nanocrystallites which causes the hardness falls [7]. The small grain size becomes impossible to accommodate the several dislocations which are required to form pile-up at the grain boundary [19]. The hardness of Fe-50%Al coating after heat treatment is higher than before heat treatment. It linearly increases with increasing milling time. This can be attributed to the existence of FeAl intermetallic compound. The increase in the hardness of coated layer after heat treatment is also due to a denser structure. As shown in SEM images (Figure 3a-d), the microstructure of coating exhibits finer grain and low porosity. It may cause by the Al diffusion in the Fe matrix during heat treatment.

Figures 5 and 6 show the Vickers indented morphologies of Fe-50%Al coating before and after heat treatment, respectively. The result shows that the Fe-50%Al coating before heat treatment is ductile. It is indicated by the absence of crack in the corner of indented samples after Vickers indentation by using a load of 500 kgf for 15 second. It could be related to that the elements of Fe and Al are not reacted after the coating process using MA (as shown on XRD analysis in Table 1). The hardness of Fe-50%Al coating after milling higher than the hardness of Fe (90 Hv) [2] and pure Al (17.03 Hv) [20]. In this case, the difference in the hardness may be attributed to the grain size [18] of coating. The optical microscope images of Fe-50%Al coating after heat treatment indicate the existence of irregular cracks on the indented section (Figure 6). This is due to the formation of FeAl intermetallic phase after heat treatment (as shown on XRD analysis in Table 2). Risanti et al. show that intermetallic phase is brittle at the room temperature [21]. Study on WC-FeAl composite by Furushima et al. revealed that the Vickers hardness is influenced by the generation of the h-phase [22]. The hardness value of Fe-50%Al coating in this study is smaller than the FeAl intermetallic (470 Hv) as presented by Ozaki and Kutsuna [23]. It may be influenced by the non uniform particle size distribution and the presence of pores in the coating (as shown on the SEM image in the Figure 3). The result strongly suggests that the hardness value is affected by the phase, grain size, and porosity of coating layer.

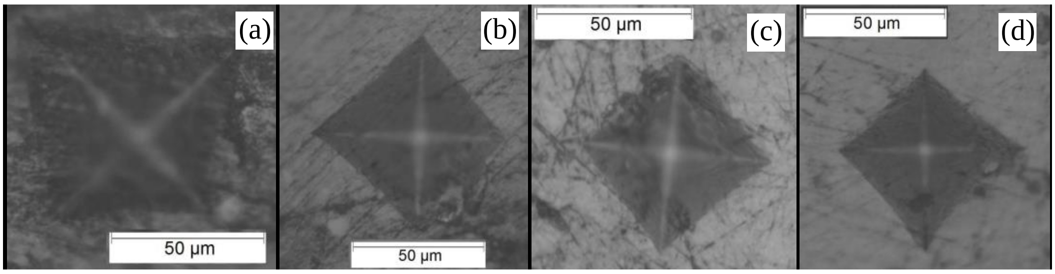

Figure 5. Optical microscopy images showing the indentation impressions produced with a load of 500 kgf for 15 second in the Fe-50%Al coating after milling time for (a) 30 min, (b) 60 min, (c) 120 min, and (d) 180 min.

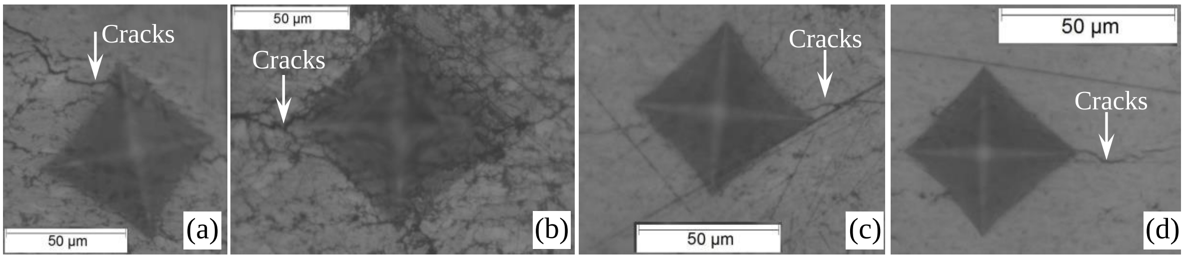

Figure 6. Optical Microscopy images showing the indentation impressions produced with a load of 500 kgf for 15 second in the Fe-50%Al coating after heat treatment at 600ºC with milling time for (a) 30 min, (b) 60 min, (c) 120 min, and (d) 180 min.

CONCLUSIONS

Mechanical alloying technique was used to deposit Fe-50%Al coating on the surface of low carbon with various milling times. The structure of coating layer was strongly influenced by the milling time during mechanical alloying process. It also affected the coating hardness. Increasing milling time caused the reducing size of elongated lamellar structure in the coating and the increasing thickness and hardness values of coating. After heat treatment, the hardness value of all samples was increased and higher than that of after milling. It was caused by the formation of FeAl intermetallic and a denser coating structure. In this study, the morphology and phase of coating were strongly correlated to the hardness values.

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