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Microstructure transformation of Cr-Al-BN coating on low carbon steel prepared by ball milling method

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Abstract: Cr-Al-BN powder was successfully applied as a coating agent on low carbon steel substrate by mechanical alloying method. The addition of BN as a doping material is considered as a feasible option because of excellent thermal and chemical stability presented by BN. In this paper, the compositions of Cr-Al-BN are varied Cr49.5-Al49.5-BN, Cr48.5-Al48.5-BN3, and Cr47.5-Al47.5-BN5. Each powder was milled by shaker mill for 2h in a stainless steel chamber with stainless steel balls to powder ratio 10:1. Subsequently, each powder compositions were mechanically alloyed onto substrate surface in air for 2h. The 2h heat treatment at temperature 800°C was conducted to each coated sample in vacuum furnace. In order to achieve the characteristics of phase composition and microstructure of the coating before and after heat treatment, XRD and Optical Microscope were performed, while the automatic microhardness tester was carried out to get the hardness of coating layer. The oxidation behaviour of coated substrates was also studied by heating treatment at 800°C for 8 cycles where each cycle is 20 h. The results show that the ball milling induces the formation of homogeneous Cr-Al-BN coating structure with a thickness of about 62.5 µm. The optimum coating hardness and oxidation resistance was achieved by Cr47.5-Al47.5-BN5 coating composition. The addition of higher concentration of BN increases the tendency on the formation of intermediate phase of Cr₂B and Cr₂N. The detailed result of this study is shown in this paper.

1. Introduction

Low carbon steel with (0.025-0.25% C) nowadays has become a famous commodity among all industries bearing to its performance and market opportunity [1]. Low carbon has good flexibility, easy to shape and low cost. However, low carbon steel manufactures encounter limitation in term of oxidation. Hence, coating is the common way to prevent this problem [2]. Various researches and developments have been conducted in order to improve the properties of low carbon steel.

Al-Cr based-mixtures are the most common element for protective layer. Various elements have been co-doped with Al-Cr to get strong and heat resistant coating. The Cr-Al-N, Cr-Si-N, and Cr-Al-Si-N have been successfully developed and the result shows the improvement of hardness level reduction with respect to friction coefficient [3]. Furthermore, previous study of Cr-Al-N, Ti-C, and Cr-B-N coating



also confirmed that the coating composition successfully improve the hardness and wear resistance of the substrate [4]. The mechanical alloying process is a method for producing composite metal powders. Mechanical alloying is an entirely solid state process, permitting dispersion of insoluble phases such as refractory oxides and addition of reactive alloying elements such as aluminum and titanium. The process was originally invented for the production of oxide dispersion strengthened (ODS) Ni-base super alloys and later extended to other ODS alloys [5-9]. Recently, many studies about coating are carried out with mechanical alloying method [10-12]. Mechanical alloying can be used to synthesize nanocrystalline-Si powder with different bandgaps, [10] and soluble catalyst systems may be rapidly produced by mechanical alloying [12].

Prior to this research, an Al-Cr base coating with addition of Tungsten (W) by mechanical alloying technique demonstrated that the mechanical alloying successfully subjected Al-Cr-W coating onto low carbon steel [13]. Presently, Boron Nitride (BN) was investigated as alternative composite in order to improve the properties of Al-Cr alloy. Heat treatment was carried out to refine the microstructure of the coating. The metallographic profiles of the coating before and after heat treatment were studied by means of XRD and Optical Microscope. Meanwhile, the hardness and oxidation resistance test were conducted to get the detail properties of the coating.

2. Experimental Details

The experiment was intended to investigate the physical properties from Cr-Al-BN varied coating composition. The substrates prepared were low carbon bar with dimension (10x10x3) mm which was polished using abrasive SiC paper up to 1500 grit. Before the coating deposition applied, the substrate was ultrasonically cleaned in methanol solution and dried by hot flowing air.

The followed step was processing the high purity Cr-Al-BN mixture. The Cr-Al-BN powder were mixed in stainless steel vial using shaker mill for 1 h with powder to steel balls ratio 1:10. The composition of Cr-Al-BN mixture was varied Cr49.5-Al49.5-BN1, Cr48.5-Al48.5-BN3, and Cr47.5-Al47.5-BN5 at%, respectively, where the total mass of each variation was 4 gr. The carbon steel specimens were then charged into the vial containing the Cr-Al-BN mixture. The specimen was mechanically alloyed under air atmosphere for 2 h. The deposition of coating powder on the steel surface is the result of collision between steel balls and powder on low carbon surface.

The annealing treatment was carried out at 800°C to improve the coating microstructure. The coated specimens were heated in vacuum atmosphere of 5.6 Pa for 2 h. Thereafter the specimens were then allowed to cool down to room temperature. The morphology of the coated sample was characterized by optical microscope (Best Scope). Analysis of phase composition of coating was conducted by X-ray diffraction (Smart Lab Rigaku) with Cu K α radiation at 40 kV and 30 mA. The hardness of coating layer was measured using automatic microhardness tester. The cyclic oxidation test was also performed in order to measure the oxidation resistance of coating layer in high temperature condition. The measuring of dimensional and weight of the samples were prepared using digital calipers and electronic balance with a sensitivity of 1 x 10⁻⁵ g prior to oxidation cyclic process. Each sample was heated at 800°C for 20 h. The weight of each sample was measured after the oxidation test, including the weights of spalled oxide. The sample was then oxidized again for 20 h. This procedure was repeated for up to 8 times and each cycle was 20 h.

3. Results and Discussion

The cross-sectional optical images of Cr49.5-Al49.5-BN1, Cr48.5-Al48.5-BN3, and Cr47.5-Al47.5-BN5 coatings before heat treatment are shown in figures 1 respectively. The figures exhibit that the coatings were successfully deposited on the low carbon steel surfaces. Mechanical alloying was effectively used to cover all substrate although the density of the coating is subsided bearing to the increment of BN concentrate. The Cr49.5-Al49.5-BN1 composition shows denser structure followed by Cr48.5-Al48.5-BN3, while the Cr47.5-Al47.5-BN5 looks more porous.

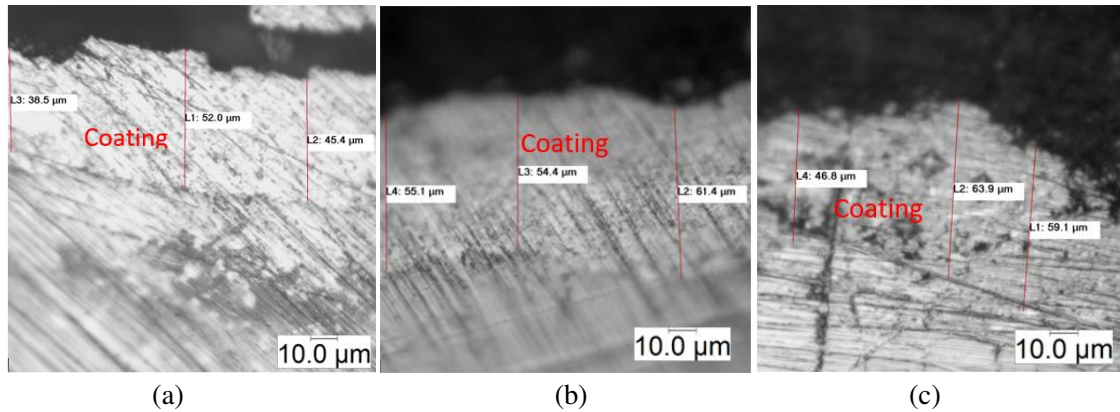


Figure 1. cross-sectional optical images of (a)Cr49.5-Al49.5-BN1 (b) Cr48.5-Al48.5-BN3 (c) Cr47.5-Al47.5-BN5 before heat treatment

Figure 2 shows the cross-sectional optical images of Cr49.5-Al49.5-BN1, Cr48.5-Al48.5-BN3, and Cr47.5-Al47.5-BN5 coatings after heat treatment at 800°C for 2 h.

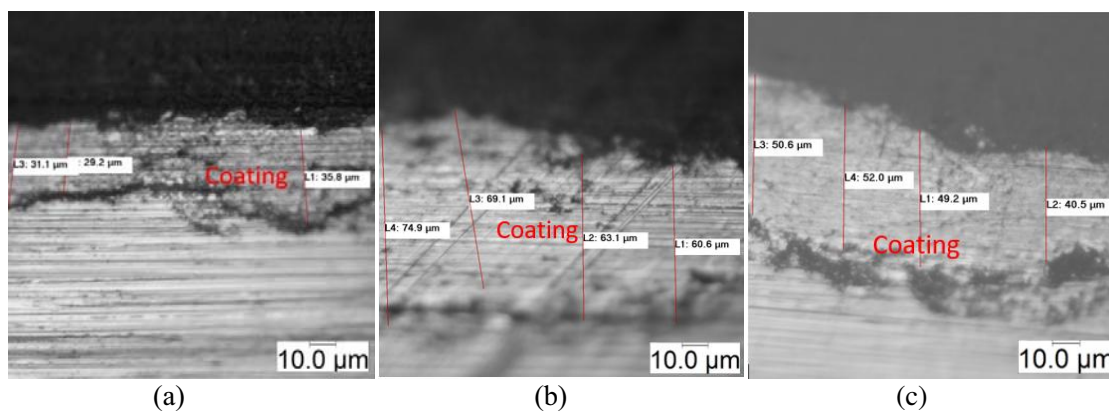


Figure 2. cross-sectional optical images of (a) Cr49.5-Al49.5-BN1; (b) Cr48.5-Al48.5-BN3; (c) Cr47.5-Al47.5-BN5 after heat treatment

Figure 3 presents the X-ray diffraction patterns of Cr49.5-Al49.5-BN1, Cr48.5-Al48.5-BN3, and Cr47.5-Al47.5-BN5 coating before heat treatment. The XRD analysis reflects that all elements in the coating remain in their original phase. Diffraction peak of Cr, Al and BN are detected on all sample, even though the diffraction peak of Al for Cr47.5-Al47.5-BN5 sample is relatively lower than that of the other two. There is no trace of intermediate phase, as usually seen for the mixture which experience mechanical milling. Interestingly, the first Al peak around 38° is not observed for Cr49.5-Al49.5-BN1 sample.

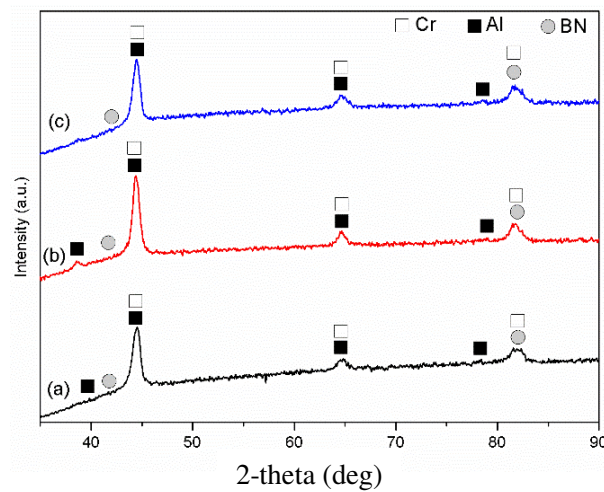


Figure 3. X-ray diffraction patterns of (a) Cr_{49.5}-Al_{49.5}-BN₁, (b) Cr_{48.5}-Al_{48.5}-BN₃, and (c) Cr_{47.5}-Al_{47.5}-BN₅ coating before heat treatment

Figure 4 shows X-ray diffraction patterns of Cr_{49.5}-Al_{49.5}-BN₁, Cr_{48.5}-Al_{48.5}-BN₃, and Cr_{47.5}-Al_{47.5}-BN₅ after heat treatment. The XRD patterns show that there is no trace of initial BN, implying the dissolution of BN species within the sample. The initial dominant phases were likely to be AlCr₂ and Al₈Cr₅. Adding 1 at% of BN did not give any additional phase. When the concentration of BN was increased to 3%, the Al₈Cr₅ was suppressed to some extent, yet still did not induce the formation of additional phase. Interestingly, the additional phases containing B and N element occurred when the BN concentration was increased to 5%. It is likely that the BN species experience bond cleavage during annealing. There is no single trace of Al₈Cr₅, while the new phase of Al₅Cr occurred. The BN bond cleavage is likely followed by the binding of Cr species in Al₈Cr₅, leading to the formation of Cr₂B and Cr₂N. As a result, the remaining Al-Cr phase experienced Cr deficiency which was resulted to the formation of Al₅Cr. As for AlCr₂, the existence of bond cleaved-BN species seems had no effect on the stability of AlCr₂ phase.

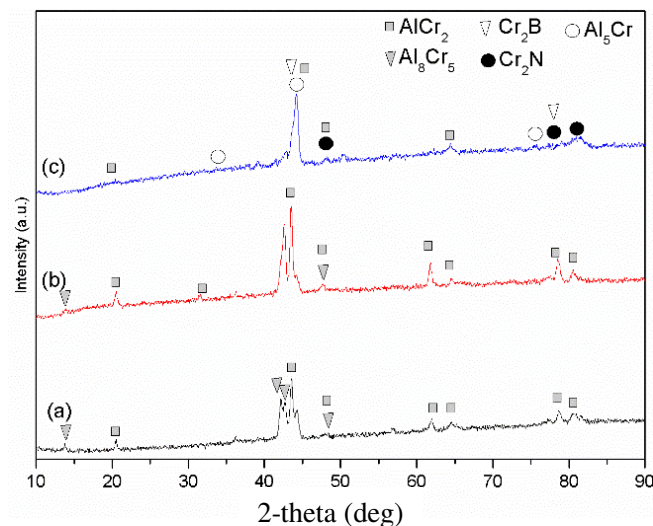


Figure 4. X-ray diffraction patterns of Cr_{49.5}-Al_{49.5}-BN₁, Cr_{48.5}-Al_{48.5}-BN₃, and Cr_{47.5}-Al_{47.5}-BN₅ after heat treatment.

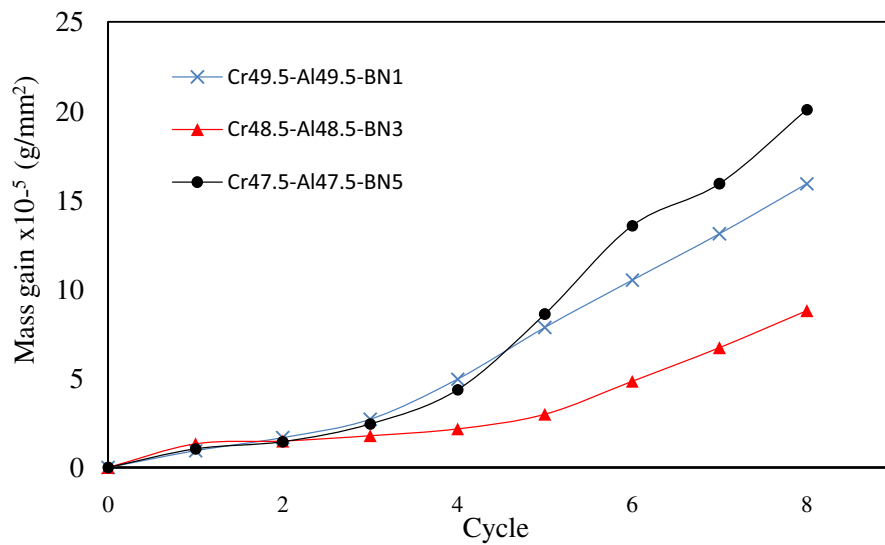


Figure 5. Cyclic oxidation curve of Cr49.5-Al49.5-BN1, Cr48.5-Al48.5-BN3, and Cr47.5-Al47.5-BN5 coating after heat treatment

Figure 5 shows cyclic oxidation curve of Cr_{49.5}-Al_{49.5}-BN₁, Cr_{48.5}-Al_{48.5}-BN₃, and Cr_{47.5}-Al_{47.5}-BN₅ after heat treatment. The mass gain in this study represents the formation of oxide materials on the coating surface. The good oxidation resistance is indicated by the lowest mass gain of the materials. The most oxidation resistance was attained by Cr_{48.5}-Al_{48.5}-BN₃.

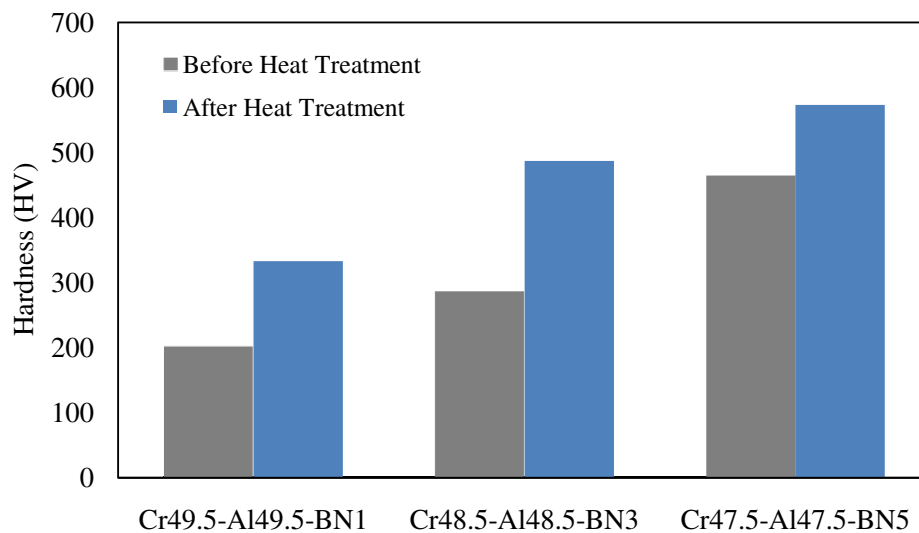


Figure 6. Hardness of Cr49.5-Al49.5-BN1, Cr48.5-Al48.5-BN3, and Cr47.5-Al47.5-BN5

Based on hardness data of Cr_{49.5}-Al_{49.5}-BN₁, Cr_{48.5}-Al_{48.5}-BN₃, and Cr_{47.5}-Al_{47.5}-BN₅ (Figure 6), the hardness level is increased by heat treatment process. The more BN additions in the coating materials, the higher hardness level can be achieved.

4. Conclusion

The coating of three different Cr-Al-BN compositions has been successfully deposited on low carbon steel using mechanical alloying technique. From the observed result, it can be concluded that the void and crack were formed in all sample after heat treatment given. The highest BN concentration (Cr47.5-Al47.5-BN5) induces the formation of Al_5Cr , Cr_2N and Cr_2B . The increase in BN concentration was also responsible to the increase of hardness. Optimum hardness was achieved from Cr47.5-Al47.5-BN5 coatings, while the less the BN concentration the less hardness level established. However, the most oxidation resistance was attained by Cr48.5-Al48.5-BN3.

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