



Surface hardness improvement of Al via inserting ceramic powders by ultrasonic mechanical coating and armoring with subsequent annealing



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ABSTRACT

In this study, ultrasonic mechanical coating and armoring (UMCA) with subsequent annealing is applied to improve surface hardness of Al. The micro-scaled CuO powders are bombarded and inserted into Al by UMCA, leading to the increment in surface hardness by 6 times. The grain refinement, work hardening and dispersion hardening are contributed to surface hardening. Moreover, the reaction temperature between CuO and Al can be reduced by severe UMCA process. Thus, after UMCA and subsequent annealing at 500 °C, the surface hardness can be increased by 10 times, which can mainly refer to the dispersion strengthening caused by transformed nano-size Cu₂O.

1. Introduction

Aluminum is widely used in industry for structural and functional purposes [1]. However, aluminum surface itself possesses low hardness and poor wear resistance [2]. Many surface modification techniques have been developed and widely studied to improve its drawbacks [3–9]. Nevertheless, most treatments exist some technical obstacles or limitations, such as complicated preparation, high operating temperature, chemical pollution, etc. Some of the surface treatments are applied to coat other materials, forming aluminum-metal matrix composite to reach the purpose of reinforcing the surface [10,11]. The coating usually measures a few micrometers in thickness, and there is inevitably a sharp interface formed between the Al matrix and hard coating layer. Recently, a solid surface modification, ultrasonic mechanical coating and armoring (UMCA), has been demonstrated to be a simple and rapid way to achieve better surface properties of aluminum [7]. Komarov et al. [12] have used UMCA to insert TiN powders into Ti, Al, and steel, and observed that the microhardness was raised by the values of 76%, 92% and 99%, respectively. Later, they also conducted UMCA on the stainless steel substrates with LaPO₄ powders, discovering the improvement in hot corrosion [13]. However, when it comes to the mechanical coating method, the bonding strength of powders and matrix is still being challenged but there is no literatures reported the optimization of UMCA. Thus, it is promising to find optimization methods to improve the UMCA process.

In this study, we intend to coat CuO powders onto a soft Al substrate through the UMCA process. Highly vibration-accelerated balls bombard the Al substrate and hammer the ceramic powders into the Al substrate. In order to enhance the bonding strength, subsequent annealing is also conducted. During heating, Al can react with CuO to generate the compounds which can further increase the hardness and wear resistance [14,15].

2. Experimental procedures

Commercial Al alloy, AA1050, consists of 99.5 wt% Al, and trace elements such as Si and Fe, was chosen as the substrate material in this research. Commercial CuO powders with a Gaussian powder size distribution and an average powder size around 10 ± 5 μm (98% purity, Sigma-Aldrich Co. LLC.) were used in the UMCA process. Such CuO powders were being mixed with 5 mL ethanol to form a suspension, and then spread onto the Al substrate. The vibration generator was powered by a piezoelectric motor. High-amplitude oscillations of the resonator was applied under a fixed vibration frequency of 20 kHz and vibration amplitude of 60 μm, resulting in an acceleration of the stainless steel balls (1 mm in diameter) to initiate their chaotic motion inside the chamber. The bombardment of the stainless steel balls produced strong impacts on the substrate surface. The CuO powders around 10 μm in diameter would broke apart to smaller sizes and being inserted into the soft Al substrate during UMCA. The coating and armoring treatments

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caused by the impact of stainless balls were conducted 2 min for a cycle. Each sample was being undergone ten cycles, i.e. 20 min of UMCA treatment. After completing the entire coating and armoring treatments, the samples were rinsed with alcohol to exclude those weakly-attached powders from the Al substrate. Subsequent annealing heat treatments were conducted at 200, 300, 400, 500 and 580 °C, respectively. The annealing processes were lasted for 1 h, followed by air-cooling.

The XRD analysis was performed using Bruker D8 system diffractometer with the Cu-K α radiation and a copper single crystal monochromator. The volume fraction of each phase can be calculated by the relative integral intensity. The cross-sectional microstructures of the coatings were observed by using JEOL 6330 scanning electron microscope (SEM). The cross-sectional thin foils for transmission electron microscopy (TEM) were fabricated using the dual-beam focused-ion-beam (FIB) system and examined by the FEI Tecnai F20 field emission transmission electron microscope with an operating voltage of 200 kV. The surface hardness was measured by Vicker's micro-hardness tester with 100 g loading stress and 10 s holding time. At least 20 readings were being collected and the calculated average is presented in the following.

3. Experimental results and discussions

3.1. Morphology and microstructure analysis

Figs. 1(a) and 1(b) show the SEM backscattered images (BEI) for the top and the cross-sectional views of the UMCA treated samples for 10 cycles (or 20 min). The brighter areas are coated CuO powders while the darker areas are the Al matrix. The effective depth of UMCA is about 10–15 μm and the surface roughness was seen to scattered about $\pm 1 \mu\text{m}$. Typically the surface coverage of CuO powders is around $80 \pm 5\%$, but the coverage will decrease with increasing depth. Fig. 1(c) presents the systematic XRD curves for all the samples. Ranging from the sample surface to the 5 μm deep inner area, the powder shows an average volume fraction (V_f) about $30 \pm 1\%$. The index

particularly for the sample annealed at 580 °C indicates the presence of many phases, such as Al, Cu, Al₂Cu, Al₄Cu₉ and Al₂O₃. Based on the XRD integral intensity and the extensive TEM analyses, the approximate volume fractions for various phases are calculated and summarized in Table 1. It should be mentioned that the grain size of the Al matrix after 20 min UMCA is around 50–100 nm, which is similar to our previous SMAT or UMCA studies [6,16].

Fig. 2 shows the TEM analysis for the sample annealed at 500 °C. By determination of diffraction pattern shown in Fig. 2(b), it can be determined that there are remaining CuO in the sample presented in Fig. 2(a). The typical size of remaining CuO has been decreased to 50–200 nm. Moreover, there are many nano-crystalline particles besides the remaining CuO fragments shown in Fig. 2(c), the particle size is only about 20 nm. By identifying the corresponding diffraction pattern shown in Fig. 2(d), it can be confirmed that there is the existence of reaction product, namely, the Cu₂O fine particles, which is consistent with the XRD results.

On the other hand, Fig. 3 shows the TEM results of the sample annealed at 580 °C. With the analysis of selected diffraction pattern (SAD) shown in Fig. 3(b), it can be found that there are the Al₂Cu phase (or usually termed as thermodynamically-stable θ phase) mixed with polycrystalline Al. The Al₂Cu θ phase in Fig. 3(a) is quite large, reaching 1–3 μm or larger. By examining the bright field image shown in Fig. 3(c) comparing with dark field images shown in Figs. 3(d) and 3(e), using the DF1 and DF2 reflections which are being circled in Fig. 3(b), respectively. It can be concluded that Al₂Cu is formed besides Al with many Al nano-particles presence inside the bulky Al₂Cu θ phase. It is similar to the Al–Al₂Cu eutectic structure reported in the literature [17]. Furthermore, Al₂O₃ precipitations are also found in the sample annealed at 580 °C. Fig. 4(a) shows the bright field image and Fig. 4(b) shows the corresponding diffraction pattern. It can be observed that the main diffraction pattern is composed by Al matrix with beam direction along [011] as well as the Al₂O₃ multicrystalline diffraction ring. By the careful determination of the diffraction ring, the phase of Al₂O₃ is found to be γ phase. With the dark field images shown in Fig. 4(c) and (d), the Al matrix and the γ -Al₂O₃ can be clearly examined, respectively. These

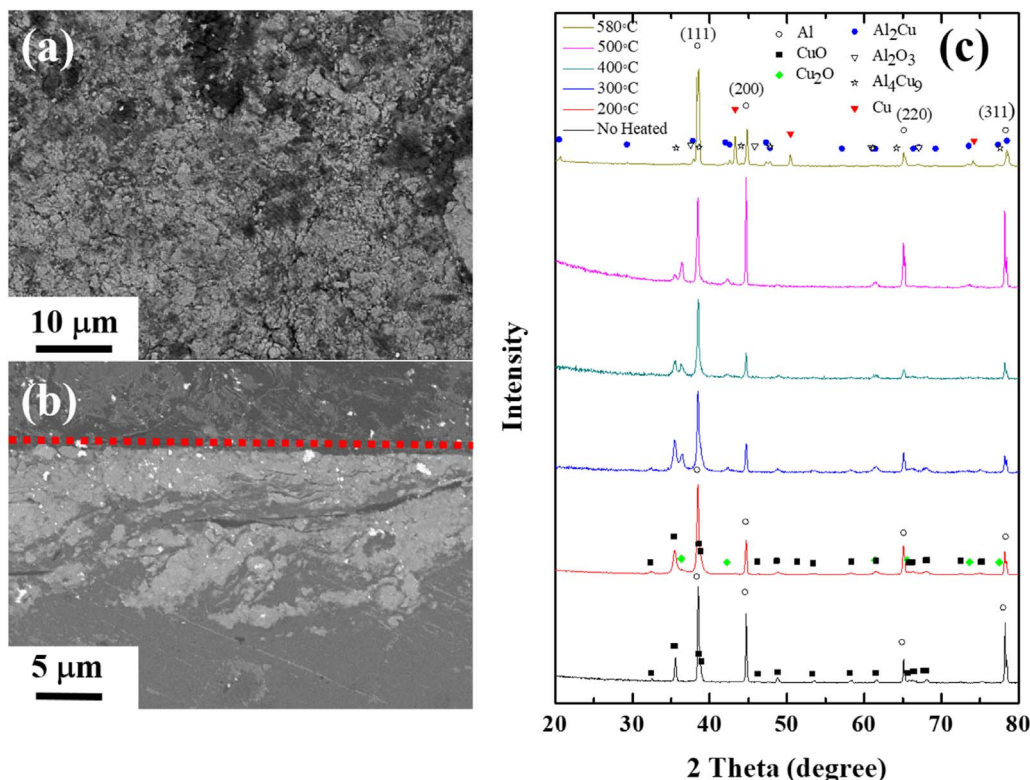


Fig. 1. The SEM backscattered electron images of the (a) top view and (b) cross-sectional view of the UMCA samples. Note that the red dotted line indicates the surface of the sample, the upper part is the resin to mount the vertical sample. (c) The XRD results of the UMCA samples, as well as the samples subject to UMCA + annealing at 200 to 580 °C. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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