



Effects of Cu content on the microstructures and properties of Cr–Cu composite coatings fabricated via mechanical alloying method



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ABSTRACT

Cr–Cu coatings with a composite structure were fabricated by means of mechanical alloying. Powders with various Cr–Cu ratios were used to fabricate coatings. Microstructures and chemical compositions of as-synthesized coatings were analyzed by SEM and EDS. Phases and microstructural features of ball milled powders were identified by XRD and SEM. The results indicated that large amount of initial Cu powder addition led to the excess of pure Cu after Cu–Cr mechanical alloying, which prevented particles from being fractured and thus resulted in the increase of surface roughness. Coatings with a multilayered structure could be detected in the cross-section micrographs. The formation mechanism of the coating was discussed. Adhesion behaviors of the coatings were determined through the combination of scratch tests and SEM equipped with EDS. Mechanical properties such as microhardness and friction and wear resistance were also tested. The test results provided an evidence of the surface reinforcement of as-synthesized coatings. Taking into consideration surface roughness, coating adhesion and mechanical properties including microhardness and friction and wear resistance, the optimal Cr–Cu ratio of starting powder was about 13:7 at the selected mechanical alloying processing parameters.

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1. Introduction

Pure copper possesses favorable ductility, electrical conductivity and thermal conductivity; thus it has been extensively utilized in electrical, electronic and plastic manufacturing industries [1]. However, the poor strength at both room and elevated temperatures restricts the application of pure Cu where both attractive mechanical property and electrical conductivity are required. In order to enhance the mechanical properties of Cu, Cu alloys are fabricated. Cu–Cr alloy is one of the significant systems due to the high electrical conductivity and high mechanical strength [2–4]. The fabrication of Cu–Cr alloy is difficult through traditional method due to the total absence of solubility between Cu and Cr under the equilibrium condition [3]. Non-equilibrium processing method, such as mechanical alloying (MA), has been applied to synthesize Cu–Cr supersaturated solid solution [4,5]. The improvement of surface mechanical properties could fulfill most application requirements. Preparation of reinforcing coating is a conventional and significant surface modification method, and it would be interesting to fabricate X–Cu (an immiscible system in which X is the matrix) composite coating on pure Cu. Recently, surface mechanical attrition treatment (SMAT), based on MA method, has been developed for the surface modification of bulk materials. And it has been successfully used to obtain nanostructured surface layers [6–13]. MA method has also been used to fabricate surface composites [14–16], through which brittle ceramic reinforcing

particles are implanted in the surface layer under continuous ball collisions and thus result in the enhancement of surface mechanical properties. MA has the potential of producing coatings with various structures through the selection of powder system [17–22]. Fabrication of coatings using MA method is a process including simultaneous substrate plastic deformation, particle mechanical milling and alloying, as well as coating deposition. Also, the thickness and roughness of coatings prepared by MA can be controlled and optimized through the combinations of MA process parameters including milling intensity, the ball-to-powder weight ratio (BPR), grinding ball size and milling duration.

In the present work, MA method was applied to prepare Cr–Cu composite coatings on bulk pure copper. Cr–Cu system was selected to synthesize the coatings due to the favorable electrical conductivity and wear resistance of Cr–Cu alloy used as electric contacts [5]. The selection of milling parameters was based on the previous research [23]. The microstructures, phases, and compositions of the as-synthesized coatings were characterized. The adhesion behaviors of the coatings were determined. The microhardnesses of as-prepared coatings were measured.

2. Experimental details

In the experiments, Cr–Cu composite coatings were fabricated using a Fritsch Pulverisette six planetary mono-mill. The schematic illustration of the experimental apparatus is shown in Fig. 1a. Cr (99.9%, <45 μm) and Cu (99.9%, <75 μm) powders were used as raw materials and pure Cu plate with the dimension of 10 mm × 10 mm × 3 mm was used as the substrate. The MA processes were carried out in a standard 700 ml

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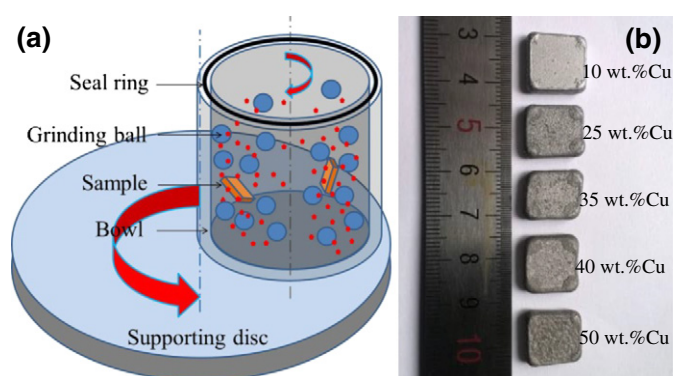


Fig. 1. Schematic illustrations of experimental method (a) and macro morphologies of surface of as-synthesized samples (b).

stainless steel grinding vial. Stainless steel grinding balls with the gross weight of 300 g were placed into the grinding vial. Grinding balls with two kinds of dimensions were used to increase the efficiency of ball-powder-substrate effects: 134 $\Phi 6$ balls (about 120 g) and 43 $\Phi 10$ balls (about 180 g). Five groups of Cr–Cu mixed powder with different weight ratio were used to fabricate Cr–Cu composite coatings: 90:10, 75:25, 65:35, 60:40 and 50:50 respectively. Samples will be identified as MA-xx, where xx corresponds to the Cu fraction in the starting powder. The total mass of powder was 30 g. No process control agent was added to the powder and the MA processes were performed in an ambient atmosphere, with the milling speed of 350 rpm and the milling duration of 7 h. The grinding vial was sealed to prevent the contamination from the atmosphere during MA treatment. Aiming to avoid an excessive temperature rising within the vial, 20 min ball milling was followed by 10 min interval.

Surface morphologies and chemical compositions of the as-synthesized coatings and cross-section microstructure features of the specimens were analyzed using a Quanta200 scanning electron microscope (SEM) equipped with an EDAX energy dispersive X-ray spectroscopy (EDX), which is used to examine the main element distributions. Phases of the ball milled powders were identified by a Bruker D8 Advance X-ray diffraction (XRD) analyzer with Cu K α radiation ($k = 0.1540598$ nm) at 40 kV and 40 mA, using a continuous scan mode.

The adhesion behaviors of as-synthesized coatings were tested using an automatic scratch instrument (WS-2006). The experimental conditions are generally showed as follows: initial load, 0.05 N; final load, 80 N; rate of load increase, 40 N/min; speed of indenter, 2 mm/min; and scratch length, 4 mm. The acoustic signal was used for the estimation of critical load, L_c . The data of L_c could be captured at the beginning of the occurrence of coating failure. It should be noted that the coating fabricated using MA method was ductile and rough, which led to the difficulties of receiving and analyzing the acoustic signals. The critical load corresponded to the load where the substrate started to be exposed. Therefore, SEM as well as EDS analyzing method was used here to give a rough assessment of the coating adhesion.

A HXS-1000A microhardness tester was used to determine the Vickers hardness of the coating layers, using a load of 0.05 kg and an indentation time of 15 s. The microhardness was tested along the profiles of the samples from the near-surface to the inner substrates. The test indents on each sample were not the same due to the different thicknesses of coatings fabricated at different powder ratio.

Sliding wear tests of the ball milled samples were conducted using a HT-500 friction and wear tester in a ball-on-disc contact configuration. The samples slid against GCr15 balls of 6 mm in diameter. The sliding wear tests were carried out in an ambient environment without lubricant, at the testing load of 1.8 N, the motor frequency of 10 Hz, the sliding speed of 560 rpm, the sliding radius of 2 cm and the testing time of 15 min.

3. Results and discussion

3.1. Surface morphologies and compositions

Fig. 1b shows macro surface morphologies of samples treated at different raw powder ratios. Apparently, all the samples were fully covered by coatings and exhibited colors different from pure Cu substrate after MA treatment. In addition, the sample fabricated at fewer Cu addition shows relatively smooth surface; thus it could be initially assumed that the weight ratio of Cr and Cu in the raw material had an effect on the surface roughness.

Fig. 2 gives surface morphologies of the samples treated at the Cr–Cu weight ratio of 90:10 (MA-10), 75:25 (MA-25), 65:35 (MA-35) and 60:40 (MA-40), respectively. The surfaces showed a tendency of densification with the increase of Cu addition in the raw materials. High magnification micrographs were provided. As shown in Fig. 2b, the MA-10 sample surface showed an equiaxed granular structure, which seemed to be made up of refined and cold welded particles. Parts of these particles were not well linked, leading to the formation of microholes in the surface, which degraded the continuity of the coatings. The high magnification image of MA-25 sample (Fig. 2d) showed the surface with high density and absence of porous. The platelet shaped particles linked together, forming the relatively smooth and continuous surface. Refined and equiaxed lamellar structure could be detected in the high magnification image of MA-35 sample (Fig. 2f), the constitutions were seamlessly linked, making up the high densified surface layer. Also, the continuous and nonporous surface of MA-40 sample could be observed in Fig. 3h. The reason of surface continuity improvement with the increase of Cu in the raw materials was that ductile Cu powder could enhance the welding ability of the whole powder system, which promoted the consolidation of refined and separated particles. Additionally, all the samples showed rough surfaces, which might be attributed to the collisions of grinding balls during MA process. The MA-35 sample showed a surface with higher smoothness in comparison with that of other samples. With a further increase of Cu in the raw powder system, surface smoothness decreased, as shown in surface morphology of MA-40 sample (Fig. 2g). Apparently, the surface of MA-40 sample was formed through the deposition of relatively large sized lamellar particles compared with that of MA-35 sample, and the formation of relatively large sized constitutions would be discussed later.

The surface microstructure features of the sample treated at the Cr–Cu weight ratio of 50:50 (MA-50) can be observed in Fig. 3. The sample exhibited a rough and hill-like surface, which was similar to the ductile fractured surface. However, there were no traces of exposed substrate to be found in the surface, which indicated that the substrate surface had been totally coated after MA treatment. In addition, although the outer layer of the coating seemed to be unfixed, it can produce stress to the inner coating layer and also protect this layer, which enhanced the adhesion between coating and substrate. The lamellar structure of the coating could be detected in the high magnification micrograph, as shown in Fig. 3b. Under the repeated ball collisions, particles especially the ductile particles were apt to be flattened and then adhered onto the substrate surface. With further ball effects, inseparable bonding between the flattened and adhered particles and the substrate was gradually formed, which led to the formation of lamellar structured coating. What's more, the rough surface was free of porous and exposed particles, which could be confirmed by the high magnification micrograph and also proved the coating to be dense and continuous.

Fig. 3c is a back-scattered electron (BSE) image corresponding to Fig. 3a, which reflects different element compositions in the coating surface. In the BSE image, the distinctions in gray scale indicated the uneven distribution of elements in the surface layer. It should be noted that although the gray scale distinctions were not apparent, there were large element composition differences in the detected surface layer. As shown in Fig. 3a, the main element content in the gray light area (belongs to the ridge of the surface) kept approaching to the raw

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