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Evaluation of Nanostructured coating layers formed on Ni balls during mechanical alloying of Cu powder

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ABSTRACT

A novel coating method utilizing mechanical alloying was used to deposit a nickel–copper solid solution on the surface of nickel balls in a planetary ball mill. Mechanically milled copper powders were deposited by cold welding on the activated surface of the nickel balls. The impact of the nickel balls causes surface wear, leading to formation of mixed layered particles of copper–nickel and rewelding. In addition, diffusion takes place through the coating layer to form a Ni–Cu solid solution. The hardness of the coating reached a threefold increase ($HV_{0.01}$ 594) in comparison with the substrate hardness. Microstructural characterization of the coating surface performed using an optical microscope, SEM, and EPMA indicates that, with appropriate processing conditions, a thick, fully dense coating can be metallurgically bonded to the nickel balls. XRD results revealed the presence of a solid solution and nanocrystalline structure.

1. Introduction

Mechanical alloying (MA) is a powder processing technique used to synthesize a variety of non-equilibrium and equilibrium materials starting from blended elemental and compound powders at room temperature in a ball mill [1]. The technique involves repeated cold welding, fracturing and rewelding, resulting in the formation of refined powders of homogeneous chemical composition. In ductile systems, the impact of the grinding balls causes the metal powders to flatten and get work harden. The flattened layers overlap and form cold welds. As the process continues, the surface of the grinding balls and the inner walls of the container are continuously impacted by the balls and powder particles, resulting in coating by the milled powder [2–4]. While a thin layer is beneficial in preventing excessive wear of the grinding medium, a thick layer results in structural inhomogeneity and it is difficult to detach from the surface. On the other hand, the process can also be used to produce surface coatings on ball milled components [5–10].

Surface mechanical attrition treatment (SMAT) technique is a new modification of mechanical alloying to obtain a nanostructured surface layer on the sample [11–13]. In this technique, the surface layer can be subjected to repeated multi-directional impacts of balls which consequently refine the coarse grained structure into a nanometer scale. It is well recognized that SMAT can be applied to prepare coatings. From the literature available it is apparent that very few attempts have been made to study coatings on milling balls.

Mechanical alloying has been employed for the fabrication of TiN, Ti–Al, Ni–Al and Fe–Al coatings [8,9]. Nevertheless, Ni–Cu system has not received much attention as a coating. Nickel and copper have identical crystal structures (face-centered cubic) and almost the same lattice parameters (approximately 2.5% misfit). Ni–Cu alloys hold immense potential as a decorative material in the industrial area, small-scale electronic devices and corrosion resistance applications [14]. However, no attempt has been made so far for the fabrication of the Ni–Cu coating using mechanical alloying technique.

In the present work, MA process has been innovatively used to structurally modify Ni grinding balls with Cu powder particles. The microstructures, phases, and composition of the coatings prepared using different MA durations were characterized. The properties of as-prepared coatings such as microhardness and thickness of coating layer are reported.

2. Experimental details

2.1. Experimental setup

Nickel grinding balls (weight 4 g, diameter 9 mm, 99.95% purity) and 3.33 g Cu powder (>99.99%, 200 mesh) were charged into the

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vial, with the ball-to-powder ratio being 30:1. The milling process was carried out in an Argon atmosphere. The milling vial was made of stainless steel of 54.5 mm internal diameter, 66.3 mm external diameter and 65.4 mm internal height. The vessels were loaded in a glove box filled with argon and sealed with an O-ring. When the chamber is properly sealed, interstitial contaminants can be reduced to a minimum during milling [10,15]. Milling was performed in a Fritsch Pulverisette 6 planetary monomill with a gear ratio of planetary wheel to system wheel of 1:−1.82. MA processes were performed at a speed of 300 rpm and grinding durations of 5, 10, 20, 60 and 120 h. In order to avoid an excessive temperature rise within the vial, 60 min ball milling was followed by a 10 min cooling interval. Finally, the milling vessel was left to cool for two hours. After processing, the powder remaining loose in the milling vial was examined by X-ray diffraction (XRD) in order to investigate the formation of the Ni–Cu phase on the surface.

2.2. Characterization of microstructure

Nickel ball specimens were cold mounted before polishing. The specimens were cut from balls using a Struers Accutom-5 cutting machine and prepared by standard metallographic procedures for characterization. The coating thickness was measured from all samples using optical and SEM microscopy and the average thickness was calculated for each sample. Geigerflex (Rigaku) X-ray diffraction (XRD) analyzer with Cu Kα radiation ($\lambda = 1.5405$ Å) at 35 kV and 15 mA was used to determine the phases of the deposited coating. A quick scan (8° per minute) was performed over the wide angle range of 30–100° 2θ. In order to determine the diffraction peaks more accurately, a slower scan rate of 0.1° per minute was used over 42–45° 2θ. The Ni crystallite size (D) was determined using the Scherrer formula [16]:

$$D = \frac{k\lambda}{\beta \cdot \cos\theta} \quad (1)$$

where k is a constant and generally assumed to be 0.9, λ is the wavelength of Cu Kα radiation, θ is half of the diffraction angle, and β is the full-width at half maximum intensity (in Radian). Specimens for metallographic examination were prepared according to standard procedures. The samples were coated with a thin layer of gold deposited by sputtering process for SEM investigation. Surface morphologies and the microstructure features of cross section of specimens were characterized using a JEOL (JCM-5700) electron microscope (SEM) at accelerating voltage of 15 and 20 kV. An Electron Probe Microanalyzer (EPMA-1720 Shimadzu) was used to examine chemical compositions. From the cross section of MAed balls, microstructural analysis and microhardness measurements were made.

2.3. Microhardness test

Vickers hardness was determined using a HM-221 type FUM microhardness tester (Mitutoyo-MicroWizhard) with a load of 0.01 kg and an indentation time of 10 s. The average of 3 indents was used to ensure acquisition of a reasonably representative value. All indents were kept away from porous locations. As the measurements were carried out in the cross-section of the coating, indents were always positioned not less than 10 μm away from the surface or the interface between the substrate and the coating.

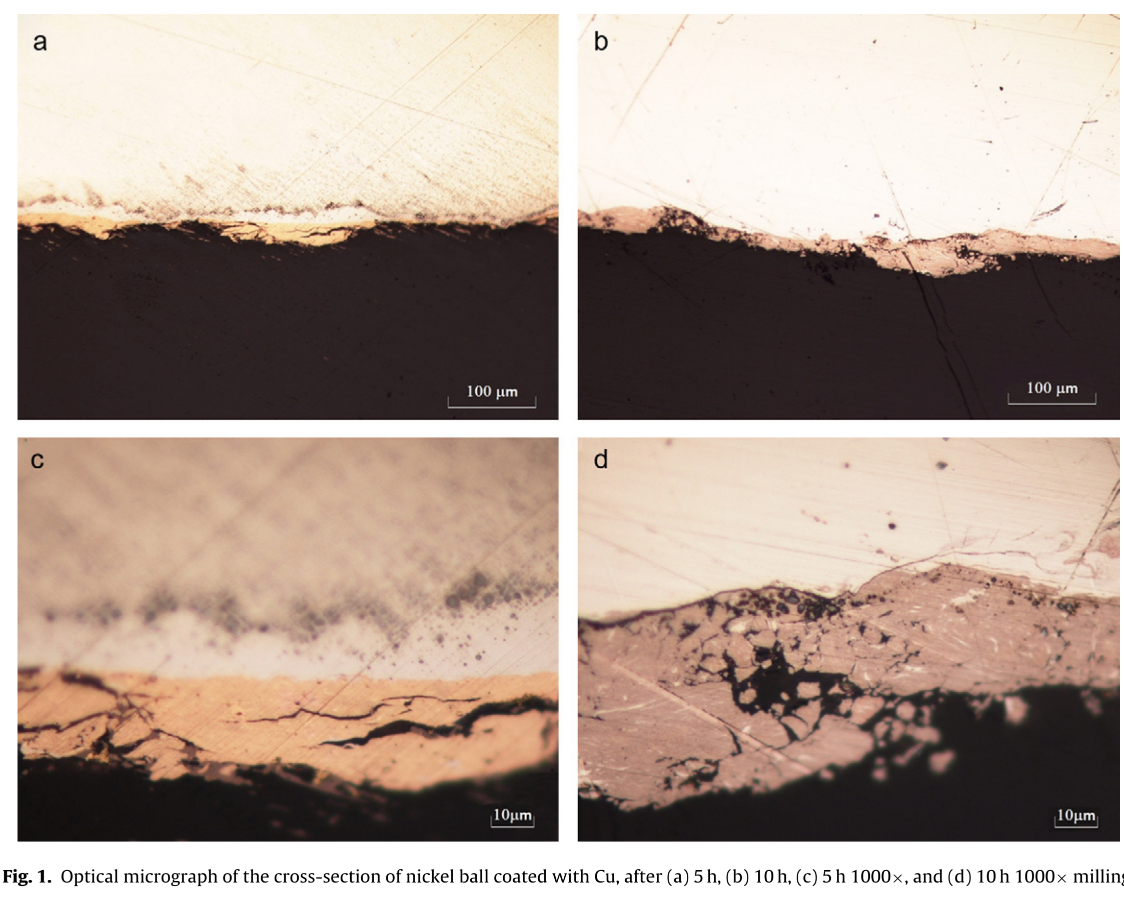

Fig. 1. Optical micrograph of the cross-section of nickel ball coated with Cu, after (a) 5 h, (b) 10 h, (c) 5 h 1000×, and (d) 10 h 1000× milling.