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## ABSTRACT

In this work, an attempt was made to further promote the wear and friction behavior of dual-scale structured Al–12 wt% Sn bearing alloys by coupling with a nano-Si@Sn composite. A so-called tri-modal Al–Sn–Si (denoted as CG-30 Si-containing alloy), which exhibited increased bulk hardness and improved distribution and refinement of the Sn phase, was produced by combining mechanical alloying and conventional powder sintering. The wear performance of the Si-containing and Si-free alloys was differentiated by performing both block-on-ring and ball-on-disk testing. Moreover, the addition of nano-Si@Sn composites to the dual-scale Al–Sn matrix resulted in significant reductions of the friction coefficient and wear volume. These improvements in wear behavior stemmed from the polishing effect of fine Si particles on the counterface, and Sn-rich clusters that acted as lubricants on the worn surface; together, the polishing effect and lubrication led to the formation of a smooth tribolayer, and hence, a decrease in the tendency for abrasive wear. Abrasive wear was also reduced by an increase in the alloy hardness with the addition of Si. As such, Sn-phase modification via Si addition constitutes a promising method for preparing self-lubricating Al-based alloys.

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## 1. Introduction

Owing to their non-toxicity as well as superior frictional and wear-resistant behaviors, Al-Sn alloys constitute important solid, self-lubricating materials for soft tribological alloy applications, and potential replacements for the Pb-containing alloys. Bearings are capable of withstanding high loads and speeds, and as such significant effort has been focused on improving the properties of Al-Sn based bearing alloys through the fabrication of nanocomposite structures. Various methods have been employed for this fabrication, including mechanical alloying (MA) [1], rapid solidification [2], physical vapor deposition [3], and severe plastic deformation [4]. The resulting nanocomposite-structured alloys exhibited excellent load-carrying capacity, as determined from the empirical Archard equation. Frequently [5-8], however, despite increases in both hardness and strength, the wear behavior of nanocrystalline (NC) alloys did not improve, and in some cases worsened, compared with those of their coarse-grained (CG)

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http://dx.doi.org/10.1016/j.wear.2015.06.017 0043-1648/© 2015 Elsevier B.V. All rights reserved. equivalents. This is attributed mainly to the low plasticity and weak work-hardening ability of the NC alloys.

Significant improvements in the wear performance of Al-Sn alloys have recently been achieved by producing, via MA, dualscale microstructures consisting of soft, coarse-grained zones and hard, ultra-fine-grained (UFG) zones [9]. The enhanced wear resistance induced by the dual-scale structure is attributed to an adequate combination of hardness and toughness, originating from optimized matching between hard and soft regions. Although the dual-scale Al-Sn alloy provides a novel method for developing bearing alloys that have high wear resistance, the presence of soft, high-ductility CG phases increases the adhesive wear, i.e., the performances imparted by the CG phases lead to increases in the adhesion component of the friction coefficient, and subsequently to a partial loss of the anti-friction characteristics of the UFG alloy [10]. The friction coefficients of our dualscale Al-Sn alloys, which contain 30-70 wt% of the CG phase, range from 0.49 to 0.54 under a force of 100 N; monolithic UFG Al-Sn alloys have a friction coefficient of 0.44. Attaining a low friction coefficient is therefore crucial to further improving the wear behavior of dual-scale Al-Sn alloys.

Previous studies have shown that a homogeneous distribution of nanoscale Pb or Sn particles in an Al matrix promotes the formation of uniform, Pb- or Sn-rich films on the worn surfaces,



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thereby resulting in significant reductions of the friction coefficient [1,11]. Moreover, grain size refinement also led to reduced adhesive wear of cast Al-based alloys [12]. Refining the Sn phase to ultrafine or nanometer sizes in the CG region constitutes therefore a promising strategy for further improving the frictional behavior of dual-scale structured Al–Sn bearing alloys. However, the very soft monolithic Sn phase is difficult to refine via milling. A precursor containing a refined Sn phase may be obtained by premilling this soft Sn with hard compounds, such as Si, WC, etc. [13,14]; the refined, soft Sn phase has excellent anti-frictional characteristics. Furthermore, the hard Si phase gives rise to a polishing effect on the counterface, thereby increasing the wear resistance and transition load [15].

In the present study, MA is used to synthesize nano-Si@Sn composites with Si nanoparticles tightly wrapped and embedded in the nano-Sn phase. The nano-Si@Sn composite is added to the dual-scale Al–Sn matrix in order to replace CG Sn. The objective is to create a designed tri-modal Al–Sn–Si composite structure that improves the wear performance. Furthermore, the role of nano-Si@Sn composites in this improvement is investigated.

## 2. Experimental

The starting Al, Sn, and Si powders (200 mesh size, 99.5% purity) were supplied by Guangzhou XinBo Co., Guangzhou, China; Sinopharm Chemical Reagent Co., Shanghai, China, and Aladdin Chemistry Co., Shanghai, China, respectively. Details of the preparation of the NC Al-12 wt% Sn powder and the dual-scale structured Al–Sn alloys are provided elsewhere [9]. In that study, the CG regions of the dual-scale structured alloy were produced by preparing mixtures of coarse Al and Sn powder. In the present study, the coarse Sn was replaced by a nano-Si@Sn composite. The alloys were prepared are as follows: (1) elemental Si powder was sealed in a stainless steel vial at a powder-to-ball ratio of 1:15, and milled for 4 h at 1000 rpm in a QM-3C shaker mill; (2) the milled Si powder was mixed with CG Sn powder in a weight ratio of 1:1 and then milled for 8 h at 800 rpm; this yielded a nano-Si@Sn composite powder comprised of nano-Si particles tightly wrapped in the nano-Sn phase; (3) CG Al was mixed with MA Sn-50Si (all percentages are by weight) powder in order to obtain an Al-nano-Si@Sn composite with a composition of Al-12Sn-12Si; (4) the Alnano-Si@Sn composite was mixed with the NC Al-12Sn powder in a weight ratio of 3:7; (5) a tri-modal Al-Sn-based bulk composite, denoted as CG-30-Si, was obtained by consolidating the mixtures under the same conditions reported in [9]. This alloy had a composition of 7.2 wt% MA Sn-50Si, 22.8 wt% CG Al, and 70.0 wt% MA Al-12Sn.

An X-ray diffractometer (XRD; Philips X'pert MPD) using Cu-K $\alpha$  radiation ( $\lambda$ =0.1541 nm) and a scanning electron microscope (SEM; Zeiss SUPRA 40) equipped with an energy dispersive spectroscope (EDS) were used to characterize the alloy microstructures. The micro-hardness at a load of 4.9 N was measured by using an HVS-1000 digital hardness tester; the final hardness value was taken as the average of at least seven measurements.

Wear and friction were measured under dry sliding conditions at ambient temperature and pressure using M-2000 and SFT-2M wear testers in the block-on-ring and ball-on-disk modes. During block-on-ring testing, the geometry of the test was similar to ASTM G77: The standard requires a ring diameter of  $34.99 \pm 0.025$  mm and a block specimen of  $15.75 \pm 0.13$  mm ×  $10.16 \pm 0.13$  mm ×  $10.16 \pm 0.13$  mm, but the current work used a 47 mm (diameter) × 10 mm (width) ring made of a hardened steel, containing 1.5 wt% Cr and 1 wt% C, with a hardness of 58–60 HRC, and a 10 mm × 10 mm × 3 mm samples under a fixed load, for 1 h, in contact with a steel ring rotating at a speed of 214 rpm. The samples were polished and then cleaned in

acetone in an ultrasonic bath. The wear and friction were characterized by the wear volume ( $\Delta V$ ) and wear coefficient ( $\mu$ ) that are defined as [1]

$$\Delta V = B \left[ \frac{\pi r^2}{180^{\circ}} \arcsin\left(\frac{b}{2r}\right) - \frac{b}{2}\sqrt{r^2 - \frac{b^2}{4}} \right]$$
(1)

$$u = \frac{F}{P} = \frac{M}{P \times r}$$
(2)

where *B*, *b*, *r*, *F*, *P*, and *M* are the width of the sample, width of the wear track, radius of the ring, frictional force, applied load, and frictional torque, respectively. The friction coefficient is the mean value of the friction coefficient over 1 h, on the applied load for the samples.

For the ball-on-disk testing in reciprocating motion in accordance with ASTM Standard G99-95, samples with sizes of  $\phi$  24 mm × 3 mm were positioned on the tribometer stage; 4.0mm-diameter steel balls (made of the same material as the ring) were used as the pin material. These steel balls rotated at a constant speed of 500 rpm under a normal load of 2 N. Each test was repeated three times in order to verify the reproducibility of the data. In addition, the frictional force was continuously monitored with a data acquisition system that calculated the friction coefficients during the test. Profiles of the cross-section of the worn scar were measured using a surface profilometer, in order to determine the wear volume; i.e.,

$$V = AL \tag{3}$$

where *V*, *A*, and *L* are the respective wear volume ( $mm^3$ ), crosssectional area of the worn scar ( $mm^2$ ), and perimeter of the worn track *L* (mm), respectively. Four measurements were conducted on each testing sample; the wear volume of each testing sample was taken as the average value of the data from the four measurements.

### 3. Results

## 3.1. Microstructure of the tri-modal Al-Sn based composite

Fig. 1 compares the XRD pattern of the MA-prepared Sn-50 wt% Si nanocomposite powder with that of the non-milled Sn-50 wt% Si powder. Owing to the immiscibility of the system, the pattern of the milled powder exhibits peaks corresponding to Sn and Si only. Furthermore, the fixed positions of the peaks, indicated that the solid solubility did not happen. However, the peaks corresponding to the Sn and Si phases broaden with milling time, owing to a size refinement of these phases. The Sn and Si phases have sizes of 90 nm and 60 nm, respectively, as estimated from the broadening of the XRD diffraction peaks using the Voigt method [16]. In addition, with increasing milling time, the peak intensity of Si decreases more rapidly than the Sn peak intensity. This decrease is attributed mainly to the spreading and wrapping of Sn on the surfaces of rigid Si nanoparticles, in a manner similar to that reported for the Ge–Sn systems [13]. Therefore, the intensity of the peaks arising from the internal Si nanoparticles is weakened by the external Sn coating. In fact, the ball milling results in hard Si particles of various sizes; particles of different sizes are distributed differently in the Sn matrix. The SEM image shown in the inset of Fig. 1 confirms that the 1- $\mu$ m-sized hard Si particles (dark region) are coated by the soft Sn phase (bright region). The TEM micrograph (Fig. 2) also indicates that many Si nanoparticles, with sizes  $\geq$  100 nm, are embedded in the soft Sn matrix.

Owing to the divorced eutectic and poor wetting of the Al–Sn alloy binary system, Sn is dispersed in an inhomogeneous, net-like

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