Surface & Coatings Technology 205 (2010) 506–510

Contents lists available at ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

Wear resistance of NiTi alloy after surface mechanical attrition treatment

T. Hu [a], C.S. Wen [b], G.Y. Sun [c], S.L. Wu [a,d], C.L. Chu [e,*], Z.W. Wu [a], G.Y. Li [c], J. Lu [b], K.W.K. Yeung [f], Paul K. Chu [a,*]

[a] Department of Physics & Materials Science, City University of Hong Kong, Tat Chee Avenue, Kowloon, Hong Kong
[b] Department of Mechanical Engineering, the Hong Kong Polytechnic University, Hunghom, Kowloon, Hong Kong
[c] State Key Laboratory of Advanced Design and Manufacturing for Vehicle Body, Hunan University, Changsha 410082, China
[d] School of Materials Science and Engineering, Hubei University, Wuhan 430062, China
[e] School of Materials Science and Engineering, Southeast University, Nanjing 211189, China
[f] Department of Orthopaedics & Traumatology, The University of Hong Kong, Pokfulam Road, Hong Kong

ARTICLE INFO

Article history:
Received 31 May 2010
Accepted in revised form 8 July 2010
Available online 15 July 2010

Keywords:
Nickel titanium shape memory alloy (NiTi SMA)
Surface mechanical attrition treatment (SMAT)
Ultrafine grain
Grain refinement
Wear resistance

ABSTRACT

An ultrafine grain layer consisting of nanocrystallites as well as submicrometer grains is produced on NiTi shape memory alloy by surface mechanical attrition treatment (SMAT) and the effects of the ultrafine grain layer on the tribological properties are investigated under dry sliding conditions. Compared to the coarse grain (CG) NiTi, the SMAT NiTi has smaller friction coefficients and improved wear resistance at applied loads from 5 to 15 N due to the grain refinement effect. Examination of the worn surfaces indicates that materials delamination and particles co-exist on both the CG and SMAT NiTi samples. Our results indicate that delamination is the main wear mechanism on CG NiTi whereas abrasive particles dominate the wear process on SMAT NiTi.

1. Introduction

The combination of good biocompatibility, corrosion resistance, as well as unique functional properties such as the shape memory effect and superelasticity renders NiTi SMAs useful in many biomedical applications [1]. Common medical NiTi devices include orthodontic wires, spine correction rods, bone fraction fixation plates, and staples [2–5]. Fretting can be quite severe in orthopedic applications and excessive wear between the NiTi implants and tissues *in vivo* can degrade the surface properties of the materials. In addition, surface corrosion raises health concerns because Ni ions released to body tissues and fluids can induce toxic and allergic responses [6]. Therefore, it is necessary to improve the surface properties such as biocompatibility, wear resistance, and corrosion resistance of NiTi shape memory alloys used in biomedical implants.

Among the different types of surface coatings, TiN is one of the most widely used due to its excellent biocompatibility and corrosion resistance. A variety of techniques such as gas nitriding [7], laser gas nitriding [8], ion nitriding [9], as well as nitrogen plasma ion implantation [10] have been utilized to fabricate TiN layers on NiTi alloys. However, these techniques either require high treatment temperature which may adversely affect the phase transformation of the NiTi alloy or produce TiN layers which are relatively thin and unable to resist long-term fretting and wear. A viable approach to solve these problems is to introduce a nanocrystalline and/or refined grain layer prior to nitriding. The nanocrystallites and refined grains possess high chemical activity due to the large number of grain boundaries and abundant defects such as dislocations and mechanical twins. These grain boundaries and defects enhance diffusion of nitrogen and formation of a thicker modified layer [11,12]. In addition, this nanostructured and/or refined grain surface layer is expected to reduce the required nitriding temperature and the plastic-deformation-induced nanocrystallites and ultrafine grains may increase the surface hardness and wear resistance as well. In fact, similar effects have been observed from several other metals [13–16]. In this work, an ultrafine grain layer consisting of nanocrystallites and submicrometer grains is created on NiTi by SMAT and the effects of grain refinement on the tribological properties are investigated and compared to conventional coarse grain (CG) NiTi.

2. Experimental details

The as-received Ti-Ni (50.8 at.% Ni and 49.2 at.% Ti) samples were first annealed to obtain homogeneous coarse grains. XRD measurements indicated that the as-annealed NiTi sample had the single austenite phase of which the crystal structure was cubic B2. Differential scanning calorimetry (DSC) measurement showed that the phase transformation points A_s and A_f of the annealed NiTi were

* Corresponding authors. P.K. Chu is to be contacted at Tel.: +852 34427724; fax: +852 27889549. C.L. Chu, Tel.: +86 25 52090683.
E-mail addresses: clchu@seu.edu.cn (C.L. Chu), paul.chu@cityu.edu.hk (P.K. Chu).

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doi:10.1016/j.surfcoat.2010.07.023

234.5 K and of 257 K, respectively. The NiTi samples were polished with silicon carbide paper before SMAT which was conducted at room temperature for 60 min using stainless steel balls 2 mm in diameter at a vibration frequency of 50 Hz. The detailed SMAT setup and process conducted at 234.5 K can be found elsewhere [17,18].

The microstructure of the surface layer on the SMAT NiTi specimen was characterized by transmission electron microscopy (TEM) using a Philips CM20 and JEOL 2010 operated at 200 kV. Thin foils were obtained by first polishing the corresponding surface layer and mechanically polishing the sample from the untreated side until it was about 40 μm thick. They were then electro-polished using a twin-jet technique in a mixture of 15% HNO_3 and 85% CH_3OH at −20 °C. Before twin-jet electropolishing, a thin coating of transparent loctite was applied onto the surface to prevent thinning of the impacted surface. After perforation, loctite was removed by dissolving in acetone, followed by rinsing with ethanol.

The cross-sectional hardness from the top surface to the substrate was determined on a nanoindenter (MTS Nano Instruments XP) equipped with the continuous stiffness measurement capability and a Berkovich (three-sided pyramid) indentor. Dry sliding wear tests were performed on an oscillating friction and wear tester (ST-3001, Teer Coating Ltd, UK) in a ball-on-disk configuration under dry conditions at room temperature in air. WC balls were used as the counter friction pair. The testing details are as follows. A 2 mm oscillating stroke with a frequency of 1.38 Hz and applied load ranging from 5 to 15 N were used. The wear depth and wear volume were determined after 30 min sliding. The profiles of the worn surfaces were measured using a surface profilometer (Form Talysurf Series 2, Taylor Hobson, UK) to determine the wear depth and wear volume. The wear volume was calculated by $V = AL$, where A refers to the worn area determined from the profile and L is the oscillating stroke. The morphologies of the worn surfaces and debris at different testing conditions were examined by an SEM equipped with EDS. Before SEM observation, the specimens were ultrasonically cleaned in acetone and alcohol for about 10 min sequentially.

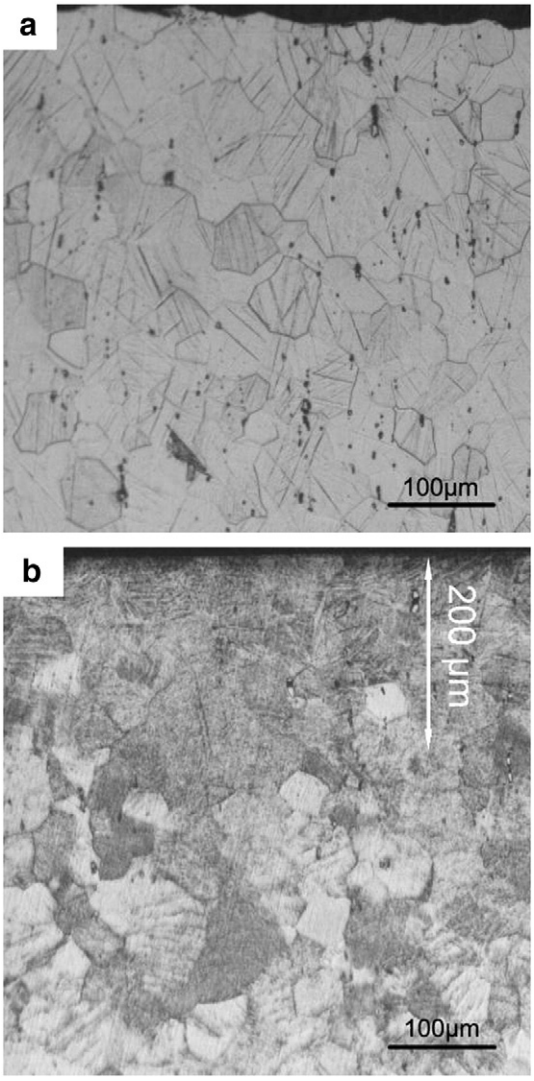

Fig. 1. The cross-sectional microstructure of (a) the CG and (b) SMAT NiTi specimens.

3. Results and discussion

Fig. 1 shows the cross-sectional microstructures of the as-received and SMAT NiTi specimens. The as-received sample has coarse grains with grain size from 40 to 80 μm. After SMAT, the coarse grains disappear and a severely deformed surface layer is produced. The thickness of this deformed layer is more than 200 μm. Fig. 2 exhibits the typical surface microstructure in the SMAT NiTi alloy. As shown in Fig. 2(a), the surface layer on the SMAT NiTi alloy contains a nanocrystalline structure. The rings in the SAED pattern indicate that these nanocrystallites are polycrystalline in nature with random crystallographic orientations. The grain size of these nanocrystallines ranges from 10 to 30 nm and the average value is about 20 nm. The grain size increases gradually with depth. At about 50 μm in depth, the characteristic microstructure shows elongated grains with micrometer size as shown in Fig. 2(b). Thereby, CG grains have been refined

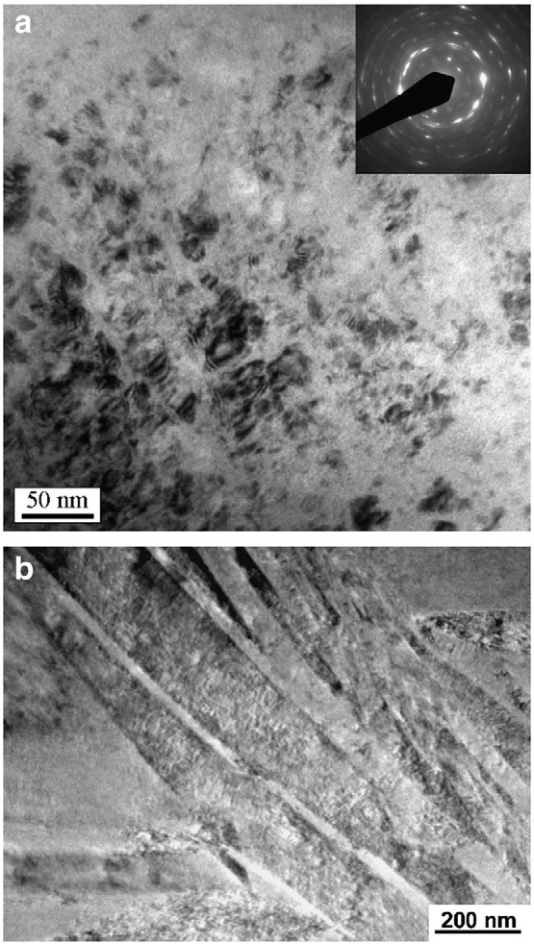

Fig. 2. (a) Bright field TEM image at a depth of about 13 μm of the SMAT NiTi, the inset is the corresponding selected area electron diffraction (SAED) pattern; (b) bright field TEM image shows the microstructure at a depth of approximate 50 μm.