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# Improved contact load-bearing capacity of ultra-fine grained titanium: Multilayering and grading

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#### ABSTRACT

The contact load-bearing response and surface damage resistance of multilayered hierarchical structured (MHSed) titanium were determined and compared to monolithic nanostructured titanium. The MHS structure was formed by combining cryorolling with a subsequent Surface Mechanical Attrition Treatment (SMAT) producing a surface structure consisted of an outer amorphous layer containing nanocrystals, an inner nanostructured layer and finally an ultra-fine grained core. The combination of a hard outer layer, a gradual transition layer and a compliant core results in reduced indentation depth, but a deeper and more diffuse sub-surface plastic deformation zone, compared to the monolithic nanostructured Ti. The redistribution of surface loading between the successive layers in the MHS Ti resulted in the suppression of cracking, whereas the monolithic nanograined (NG) Ti exhibited sub-surface cracks at the boundary of the plastic strain field. Finite element models with discrete layers and mechanically graded layers representing the MHS system confirmed the absence of cracking and revealed a 38% decrease in shear stress in the sub-surface plastic strain field, compared to the monolithic NG Ti. Further, the mechanical gradation achieves a more gradual stress distribution which mitigates the interface failure and increases the interfacial toughness, thus providing strong resistance to loading damage.

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### 1. Introduction

Materials with high contact damage-resistance are extensively required in aerospace and automotive industry, microelectromechanical systems and devices, cutting tools and bulletproof vests [1,2]. An approach to improve surface damage resistance is to design surfaces with tailored gradation in composition, microstructure and elastic/plastic properties [2,3,4]. Such design allows the redistribution of thermal and/or mechanical stresses [5,6], elimination of interface-induced stress concentrations [7], and reduction in the local crack driving force [8,9]. Over the past two decades, significant progress has been made in synthesis and fabrication of materials with elastic/plastic gradations over multiple length scales. Elastically graded materials (EGMs), where the materials have gradient in elastic modulus as a function of depth beneath the surface, were synthesized by controlled infiltration of aluminosilicate or oxynitride glass into polycrystalline ceramic matrix, which offered superior resistance to contact damage than either constituent ceramic matrix or glass [9,10,11]. Plastically graded materials (PGMs) were produced by increasing or decreasing the grain size within the nanocrystalline or microcrystalline range to create a linear gradation of yield strength as a function of depth below the material surface according to classical Hall–Petch effect [3]. The benefit of the gradient effect on the stress–strain and deformation response under normal indentation have been demonstrated by analytical [12], computational [13,14,15] and experimental studies [16,17].

In our prior work [18], we extended the EGM/PGM concept to design a multilayered hierarchical structure (MHS) on Ti. By the application of Surface Mechanical Attrition Treatment (SMAT) [19] to cryorolled Ti, a three-layered structure formed consisting of an outer amorphous/nanocrystallite (A/NC) layer, an inner nanograined (NG) layer and ultrafine-grained (UFG) core [18]. Nanoindentation through the cross-section of the MHS Ti revealed a gradual decrease in hardness and modulus within and between each successive structure characteristics and the design principle found in natural systems, such as fish armour [20,21]. The gradual transition in structure and properties, pore- and crack-free nature and the inherently damage tolerant top A/NC layer on MHS Ti are expected to benefit the contact load-bearing and damage-resistance of MHS Ti.







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Studies of the effects of microstructural, compositional, and property gradients on the overall elastic–plastic response under contact loading are an area of great interest, and much progress has been achieved in the fundamental understanding of graded surface damage resistance [2]. However, systematic investigations of the multilayered hierarchical structure on indentation response, contact damage-resistance and failure of graded ultrafine-grained (UFG) metals, in particular with regards to the structural multilayering and grading, have not been investigated by multiscale experimental and computational approaches. The mechanism of the contact load-bearing response in these situations is also largely unknown.

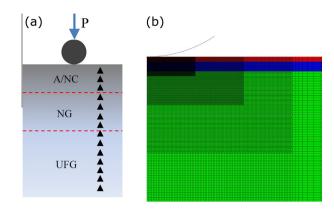
In this paper, we report the contact load-bearing capacity and surface damage resistance of MHS Ti relative to monolithic nanostructured Ti. Through experimental investigations and computational simulations of local stress and strain distributions, the mechanism of the contact load-bearing response of MHS Ti is explored. These results provide clear evidence of improved contact load-bearing capacities through structural multilayering and grading. Such information is of practical value for the design of materials with excellent contact load-bearing capacities for engineering applications.

#### 2. Experimental procedures

The MHS Ti was produced by the following experimental procedures. A commercial Ti plate (Grade 2) with 36 mm in thickness was cryogenically rolled to 5 mm with per reduction of ~2 mm. The detailed microstructure characterizations of the cryorolled Ti have been given elsewhere [22]. The cryorolled workpiece then was cut parallel to the rolling direction (RD) to a rectangular bar with dimensions of  $5 \times 5 \times 90 \text{ mm}^3$ . Subsequently, one lateral surface of the rectangular bar was subjected to SMAT. The SMAT process was performed in a low vacuum condition using hardened stainless steel balls (8 mm in diameter) at a vibration frequency of 50 Hz for 60 min. The detailed MHS process can be found in [18]. The production of monolithic NG Ti has been given in [22].

Fig. 1a shows a schematic illustration of the nanoindentation and contact load-bearing testing. Nanoindentation experiments were carried out at ambient temperature using an UMIS indentation system with a Berkovich diamond tip at a strain rate of 5  $\times$  $10^{-2}$  s<sup>-1</sup> and a maximum load of 20 mN. Before testing, the cross-section of the MHS Ti was polished to 0.5 µm diamond suspension finish. The values of the nanoindentation hardness and modulus quoted here were the average of 10 measurements on the cross-sectional surface. Before Vickers microhardness and load-bearing testing, artifacts on the surface caused by MHS process were carefully removed by polishing to 0.5  $\mu m$ dimond suspension finish (removal thickness  $< 2 \mu m$ ). Vickers microhardness testing was conducted using a microhardness tester (FM 700) under a load of 0.5, 1, 3, 5, 10 N on the MHSed surface at more than 10 points and the average values were reported here. Load-bearing tests were performed on the surface of MHS Ti and monolithic NG Ti with a spherical tungsten carbide (WC) indenter with diameter of 1.5 mm in ambient conditions. The WC indenter had an elastic modulus of 640 GPa and a Poisson's ratio of 0.26. The indenter came into contact with the specimen surface and was loaded to a maximum load of 1000 N at a loading rate of 1000 N/s. Microstructural and damage observations were conducted using a field-emission gun scanning electron microscope (SEM) Zeiss Supra 55VP operated at 10 kV and a transmission electron microscope (TEM) Jeol JEM 2100 operated at 200 kV.

A two-dimensional axisymmetric model was developed to simulate the ball indentation using Abaqus v6.10. The specimens were modelled as isotropic, elastic–perfectly plastic following the large-deformation theory. The finite element mesh contained 42163 four-node bilinear axisymmetric quadrilateral elements



**Fig. 1.** (a) Schematic illustration of the surface load-bearing and cross-sectional nanoindentation testing. The black ball represents the load-bearing testing indenter and the black triangle represents the indent of nanoindentation testing. (b) Finite element mesh of the indentation model.

(CAX4R), with a refined mesh in the indentation region (Fig. 1b). Mesh convergence was verified by comparing load-depth curves and stress contours using models with element number ranging from 11183 to 42163. A user subroutine UMAT was developed to take into account the gradation of material properties, in which the discrete and gradient model parameters were assigned to elements based on their distance from the surface (see later for further details). The ball indenter was modelled as a rigid body, and the contact between the indenter and specimen was assumed frictionless. A maximum load of 1000 N was applied to indent the samples at a loading rate of 1000 N/s. The finite element analysis (FEA) of the microindentation adopted a similar methodology to the axisymmetric nanoindentation simulations. The Vickers indenter was modelled to be a conical rigid indenter with an apex angle of 70.3° and tip radius of 3.4  $\mu$ m, which approximates a Vicker indenter tip.

# 3. Results

# 3.1. Microstructure

A cross-sectional view of the MHSed Ti surface shows three layered structure, without sharp interfaces between the successive layers (Fig. 2a). TEM analysis has revealed that the top layer, with thickness of  $\sim$ 30  $\mu$ m, was composed of a bright phase matrix and a discrete darker nanostructure (Fig. 2b). The upper right inset taken from the bright matrix region exhibited a broad diffuse halo in a selected-area diffraction pattern (SAD), which is typical of a fully amorphous phase. The SAD pattern in the lower left inset taken from the interface between the bright and dark phases clearly demonstrates the presence of a nanocrystalline phase together with the amorphous phase. High-resolution (HR) TEM images (Fig. 2e and f), taken from the marked areas in Fig. 2b further confirm the respective amorphous and nanocrystalline character of the phases. The NG layer ( $\sim$ 60  $\mu$ m thick), situated beneath the A/NC layer, consisted of nanograins (Fig. 2c). The corresponding SAD pattern shows a ring pattern, demonstrating the nanostructure has random crystallographic orientations. The size of the nanograins was in the range from 5 to 80 nm with an average size of  $\sim$ 40 nm (Fig. 2g). The UFG core is composed of ultrafine equiaxed grains with a grain size distribution of 50-250 nm (Fig. 2d and h).

#### 3.2. Mechanical gradations

Nanoindentation was used to measure the elastic and plastic mechanical properties spatially through the cross-section of the Download English Version:

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