



## Characterization of plastically graded nanostructured material: Part II. The experimental validation in surface nanostructured material

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### ABSTRACT

The computational algorithm developed in Part I was utilized to quantify the depth-dependent constitutive law of the plastically graded material (PGM) – the surface mechanical attrition treated AISI304 stainless steel. This material possesses high yield stress due to the hardened surface layers, while the necking of the hardened surface layers was retarded in a tensile test due to the more ductile core layers. A number of cross-sectional nanoindentations were conducted and the curves were processed to remove the effect of the strain rate, the tip blunting as well as the adhesion. The yield stress and the hardening coefficient could then be, respectively, calculated from the loading curvature and energy recovery ratio of a single indentation. By taking Bauschinger effect into account, the integrated stress–strain curve over the whole thickness replicates that from the tensile test well. It is noted that the subsurface layers have substantial hardening rates, which is mainly attributed to the existence of the dense nanotwins. A further discussion in this paper is addressed on the relation between the measured depth-dependent constitutive laws and the corresponding microstructures.

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### 1. Introduction

The framework for exploring the flow behaviour of the linear hardening material from the loading and unloading indentation curves is presented in Part I. Herein we present experimental results that correlate the graded mechanical properties and the microstructures. The target material, AISI304 stainless steel (304SS) sheet, is subjected to ultrasonic surface mechanical attrition treatment (SMAT). Such process provides high-speed (10 m/s; Chan et al., submitted for publication) local impact of hard spherical shots, which generate a high strain rate and large resultant strain in the subsurface layers. Technically, this process is different from the treatment used by Zhang et al. (2003). A detailed study of the motion and impact of the balls and their effect on the treatment efficiency are presented by

Chan et al. (submitted for publication). The impressive result of the treated 304SS is the excellent combination of the strength (>800 MPa) and ductility (>40%) as well as the high hardening rate. Such tensile property of the monolithic 304SS sheet was not reported in the literature. The treated material is characterized by the graded microstructures and mechanical properties, which is resulted from the distribution of the strain and average strain rate from the surface to interior layers during the treatment process (Chan et al., submitted for publication; Lu and Lu, 2003). SMAT is essentially a strain hardening process: the elastic modulus of different layers could thus be regarded as uniform and the treated material is exactly plastically graded material (PGM). Such PGM possesses large variations of yield stress and hardening rate through the thickness (1 mm), which will be demonstrated in this paper.

In order to make this paper more self-contained, the equations that will be used to analyze the indentation curves are listed as follows:

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$$\frac{C}{\sigma_r} = \left( \frac{1}{m_e E^* / \sigma_r} + \frac{1}{m_{e2} (E^* / \sigma_r)^2} + \frac{1}{m_p} \right)^{-1} \quad (1)$$

$$H = [wE^* / 18.67 \sigma_r]^{10.7} - 1.34 \quad (2)$$

$$\varepsilon_r(\sigma_r / E^*, H) = \left( K_{e1}(H)(\sigma_r / E^*) + K_{e2}(H)(\sigma_r / E^*)^2 + \frac{1}{\varepsilon_r^{RPP}(H)} \right)^{-1} \quad (3)$$

where

$$E^* = \left( \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i} \right)^{-1} \quad (4)$$

is the reduced Young's modulus;  $C$  is the curvature of the indentation loading curve;  $w$  is the recovery-absorption energy ratio;  $H$  is dimensionless hardening coefficient;  $\sigma_r$  and  $\varepsilon_r$  are the representative stress and strain, respectively. The other symbols are fitting parameters, whose magnitudes are given in Part I. As discussed in Part I, the flow behaviour of the treated 304SS is assumed to be linear hardening, which is given by

$$\sigma_r = \sigma_y(1 + H\varepsilon_r) \quad (5)$$

where  $\sigma_r$ ,  $\sigma_y$  and  $\varepsilon_r$ , respectively, pertain to the representative stress, yield stress, and representative strain.

## 2. The treated and untreated material – optical micrograph

The as-received and treated 304SS were sectioned, polished and etched to visualize the boundaries of micron-scale grains under the optical microscope. Fig. 1(a) shows the optical micrograph of the etched cross-sections of the materials before and after treatment. It is clearly observed that the subsurface region (within  $\sim 100 \mu\text{m}$  from the surface) of the treated sample possesses much greater numbers of deformation bands in each grain than the as-received material. From the selected area electron diffraction (SAED) pattern in TEM observations of the thinned cross-sectional sample, shown in Fig. 1(b), it can be confirmed that these deformation bands are mainly deformation twins. The density of the deformation twins decreases as the depth increases. A similar result can be found in the explosively loaded 304SS in Firrao et al. (2006), in which the abundance of deformation twins in the subsurface region can be observed if the impulsive force is sufficiently large. It is noted that the SMAT process applies impulsive force on the sample surface, which thus bears analogy to the explosive load. The high twinability of the 304 austenite steel stems from its low stacking fault energy ( $21 \text{ J/m}^2$ ; Murr, 1975) and the twinning mediated plastic deformation in the SMAT process is attributed to the high strain rate ( $10^4 \text{ s}^{-1}$ ; Chan et al., submitted for publication). Deformation twinning generally requires higher resolved stress at each grain (Meyers et al., 2001) and its multiplication renders higher strain hardening rates than slip does (Chichili et al., 1998; Lu et al., 2009a,b). Therefore, in the subsequent nanoindentation experiments of treated 304SS, one will note remarkable increases of the yield

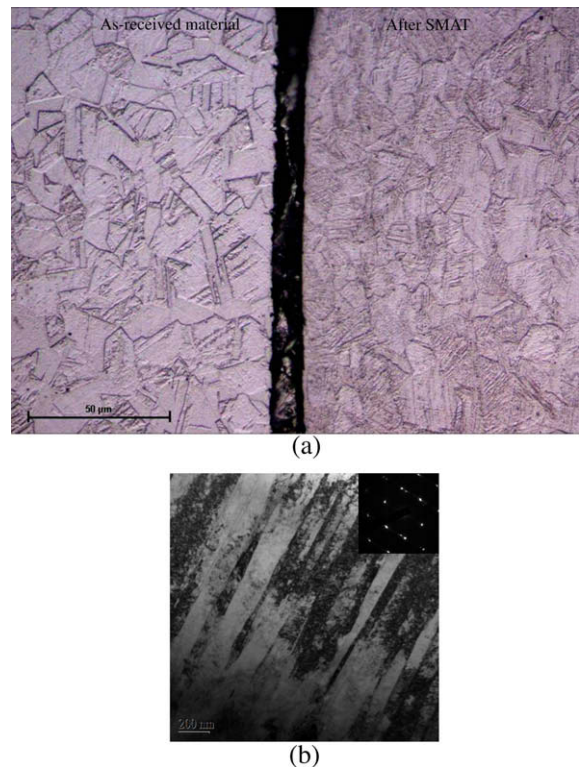


Fig. 1. Microscope images of the cross-section of the AISI 304SS before and after SMAT: (a) optical micrograph and (b) TEM image with SAED pattern inset.

stress and hardening rate, which is significantly different from the ultrafine-grained material.

## 3. The mechanical property – nanoindentation characterization

### 3.1. Experimental setup

The displacement-controlled nanoindentations were performed with the approaching speed of  $50 \text{ nm/s}$  by using a TriboScope™ nanoindenter (Hystron, USA). A Berkovich indenter was adopted. Every indentation is initiated by a contact search process, which located the surface of the sample by slowly moving the indenter tip towards the sample and monitoring the contact force. If a  $100 \mu\text{N}$  or larger contact force is recorded, the contact search finished and the sample surface is located. Afterwards, the thermal drifting is calibrated in every indentation under a very low contact force ( $100 \mu\text{N}$ ) and is automatically eliminated from the subsequent load–displacement data. At the maximum indentation depth of  $1400 \text{ nm}$ , the indenter was held for  $10 \text{ s}$  before unloading.

The indentation pattern was a  $5 \times 13 \times 40 \mu\text{m}$  array for each set of indentations. A total of three sets of indentations were conducted. For the three sets, the indentation spots closest to the boundary are, respectively,  $15 \mu\text{m}$ ,  $20 \mu\text{m}$  and  $25 \mu\text{m}$  from the treated surface. Some indentation curves were discarded since their initial parts did not

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