Acta Materialia 96 (2015) 24–36

Contents lists available at ScienceDirect

Acta Materialia

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

Formation of nano-laminated structure in nickel by means of surface mechanical grinding treatment

X.C. Liu [a], H.W. Zhang [a], K. Lu [a,b,*]

[a] Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China
[b] Herbert Gleiter Institute of Nanoscience, Nanjing University of Science and Technology, Nanjing 210094, China

ARTICLE INFO

Article history:
Received 11 March 2015
Revised 2 June 2015
Accepted 6 June 2015
Available online 14 June 2015

Keywords:
Nano-laminated structure
Surface mechanical grinding treatment (SMGT)
Strain gradient
Nanometals

ABSTRACT

A polycrystalline Ni (99.882% purity) bar sample was subjected to surface mechanical grinding treatment (SMGT) at ambient temperature. Gradient microstructures along depth from the treated surface were generated owing to a graded variation of strain and strain rates, including dislocation structures, submicron-sized structures, and nanostructures, respectively. In the subsurface layer of 10–80 μm deep, 2-dimensional laminated structures with low angle boundaries and strong deformation textures were formed of which the average thickness is ~20 nm, one order of magnitude smaller than that of the ultrafine structures in Ni induced by conventional severe plastic deformation. The extraordinary grain refinement was ascribed to the high strain rates and high strain gradients that enhance accumulation of geometric necessary dislocations with a suppressed recovery dynamics. Deformation of the nano-laminated structures is governed by dislocation slip, and supplemented by deformation twining at the nanoscale, eventually leading to fragmentation into nano-sized equiaxed grains.

1. Introduction

Plastic deformation is effective in refining grains of polycrystalline metals and alloys [1–6]. Refinement of coarse grains in metals is usually governed by dislocation activities and/or deformation twinning forming various types of boundaries that subdivide the original coarse grains, such as dislocation boundaries, low and high angle grain boundaries (GBs), twin boundaries, or phase boundaries. However, as strain reaches a certain level, structure refinement ceases, forming a saturated (or steady-state) microstructure of which both the length scale (generally in submicron regime) and boundary characteristics (mostly high angle GBs) are unchanged with a further increasing strain [6,7]. Appearance of the saturated microstructures prevents further refinement and further strengthening of materials upon plastic deformation. Although proper alloying [6–8] or twining [9–11] can reduce the grain sizes below the saturated size limit, how to break the length scale limit of the deformed microstructure for pure metals is still a challenge.

The saturation in structural evolution is related to the decreased structural stability with a reduction of structural scale. Annihilation of dislocations is enhanced as boundary spacing is reduced [8,9]. Grain boundary migration coupled with mobile triple junctions leads to structural coarsening and the removal of defects during deformation as they act as dislocation sinks [10]. It is known that high strain rates and/or low deformation temperatures are effective in reducing grain sizes to some extent [4,11]. Extraordinary grain refinement of metals has also been observed during surface plastic deformation [3,5,12–15], inside shear bands [16] and close to the hard second-phase particles [17], where high strain gradients are presented. High strain rate and high strain gradients seem effective in promoting strain-induced structure refinement. Our recent investigations demonstrated that by applying surface plastic deformation with high strain rates and high strain gradients, a pure Ni and an interstitial free steel can be refined down to a length scale as small as 10–20 nm, one order magnitude below the corresponding saturated size limits [18,19]. 2D nano-laminated (NL) structures with low angle boundaries are formed. However, several questions are open for answer, including:

(i) What is the structural evolution pattern toward nanometer scale?
(ii) How the 2D NL is formed?
(iii) How is the correlation between extreme structural refinement and the processing parameters?
(iv) Whether dislocation slip still governs the structural evolution toward nanometer scale?

* Corresponding author at: Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China.
E-mail address: lu@imr.ac.cn (K. Lu).

http://dx.doi.org/10.1016/j.actamat.2015.06.014
1359-6454/

The present investigation aims to address these questions. A pure Ni with high stacking fault energy was chosen as the experimental material to subject to surface mechanical grinding treatment (SMGT), and the depth-dependent deformation microstructure and the processing parameters (strain, strain rate and strain gradient) are quantified. Dislocation slip dominated extreme microstructure refinement mechanism is analyzed and importance of high strain rate and high strain gradient during grain refinement is highlighted.

2. Experimental

2.1. Material and processing

A polycrystalline Ni rod (10 mm in diameter and 100 mm in length) with a purity of 99.882 wt.% (Table 1) was subjected to SMGT at ambient temperature. Prior to processing, the Ni sample was annealed in vacuum at 1273 K for 10 h, forming a fully recrystallized structure with an average grain size of 550 μm (Fig. 1a). During the SMGT processing as schematically illustrated in Fig. 1b, the bar sample rotates at a velocity V_1, while a WC/Co tool tip was forced to indent into sample surface by 30 μm deep and slides along TD at a velocity V_2. As the tool tip slides from one end of the rod to the other, the sample surface is defined as being treated by one pass. In order to increase plastic strain, the sample was treated by multiple passes with an additional 30 μm in indentation depth in each subsequent pass. That means, in the second pass the tool tip indented into the sample surface by 60 μm deep, and in the third pass by 90 μm deep. The sample was treated by 1, 3, 6 and 8 passes (denoted as sample 1P, 3P, 6P and 8P), respectively. The tip indent depth in sample 8P is 240 μm. Temperature rise during the treatments is controlled by cooling oil. The tool tip radius is 8 mm, V_1 = 300 rpm, V_2 = 6 mm/min. More information can be found in previous publications [20].

2.2. Determination of strain, strain gradient, and strain rates

Plastic strain distribution in the surface layer of the SMGT Ni sample was estimated by means of an annealing twin boundary that was originally perpendicular to the tangent of the top surface. A similar estimation of the strain profile below the worn surface in a composite copper-silver solder material was performed by using an artificially inserted thin silver solder film (5 μm thick) as an internal marker [21]. The depth dependence of displacement field (y) is described by an exponential function [22]:

$$y(x) = y_s \exp(-kx) \tag{1}$$

In sample 8P, deformation-induced deflection of the twin boundary (marked in Fig. 2a) from its original position was measured in a given coordinate system (X–O–Y). The agreement between the measured data and the least square fitting of $\ln(y) - x$ implies a reasonable description of the depth-displacement dependence with Eq. (1), where k = 0.011 μm^{-1} and y_s = 5100 μm (displacement at the top surface, x = 0). Shear strain and displacement are related by:

$$\gamma(x) = -\frac{\partial y(x)}{\partial x} = 57.6 \exp(-0.011x) \tag{2}$$

As shown in Fig. 2c, the estimated shear strain in the topmost surface layer reaches ~58, and it decreases to 24–1.0 in a depth span of 80–400 μm. The strain gradient (variation of strain as a function of depth), $\frac{\partial\gamma(x)}{\partial x}$ [23], is obtained by differentiating Eq. (2):

$$\frac{\partial\gamma(x)}{\partial x} = 0.63 \exp(-0.011x) \tag{3}$$

The obtained strain gradient at the treated surface is about 0.63 μm^{-1}, as in Fig. 2d, and it drops to 0.26 and 10^{-3} μm^{-1} as depth increases to 80 and 400 μm, respectively. The strain gradient in the top 80 μm-thick surface layer is obviously higher than that of other heavy plastic deformation techniques. Taking high pressure torsion (HPT) as an example [1,7], shear strain is $\gamma = \frac{2\pi rN}{l}$ and strain gradient is: $\frac{\partial\gamma}{\partial r} = \frac{2\pi N}{l}$, where N is the revolution number, r is the disk radius and l is the thickness. For typical processing with N = 5, r = 5 mm and l = 2 mm, shear strain is about 78 and strain gradient is only ~0.02 μm^{-1}. The latter is at least one order of magnitude smaller than that in the surface layer of sample 8P.

Assuming that in a straining time (t) surface materials of the SMGT sample were sheared by a distance y_s at a linear velocity ($2\pi RV_1$), strain rate can be estimated by taking $t = y_s/(2\pi RV_1)$:

$$\dot{\gamma}(x) = \frac{\gamma(x)}{t} \tag{4}$$

As t is approximately ~10^{-2} s, the strain rate in the top 80 μm-thick surface layer is about 10^3–10^4 s^{-1}, which agrees with a previous estimation in a SMGT Cu sample (10^4 s^{-1} [20]).

The above estimation shows that the surface layer of sample 8P is deformed with a very large strain, high strain rates and high strain gradients, which drop gradually with an increasing depth. For samples 1, 3, and 6P, similar distributions of these quantities are expected but with smaller amplitudes. It is noted that the plastic strain in the top surface layer, in which stress states are more complicated, might be underestimated by simple extrapolation of the measured displacement as a function of depth in the deeper (>250 μm) subsurface layer. Besides, Eq. (2) considers only the shear along V_1, whereas contributions from compression by tool tip and shear along V_2 were less significant and neglected. Consequently, the above estimation might provide a low bound approximation, in particular for multi-passes samples.

2.3. Microstructural characterization

Microstructures of SMGT samples were characterized by using scanning electron microscopy (SEM) and transmission electron microscopy (TEM) on the SD–ND plane, i.e., the cross-sectional plane. The treated surface was firstly protected by an electro-deposited Ni coating, followed by cutting, mechanical polishing and final electrolytic polishing in electrolyte ($HClO_4$:CH_3COOH:C_2H_5OH = 1:3:4) at −10 °C. Slices of 1 mm thick were cut parallel to SD–ND plane and mechanically thinned down to 50 μm thick, followed by final thinning by using a double-jet electrolytic polishing (electrolyte: 10 vol.% $HClO_4$ + 90 vol.% C_2H_5OH, at −20 °C) supplemented with ion-milling (Gatan 691). The cross-sectional microstructures were observed in a JEOL 2010 TEM and FEI Tecnai F30 high resolution TEM (HRTEM). The boundary spacing of the laminate structures was determined by measuring the interception length along lines perpendicular (D_T) and parallel (D_L) to the extending direction, respectively. The boundary misorientation angle was determined by convergent beam electron diffraction (CBED) analysis. It includes obtaining the orientations and orientation matrix of the crystallites on both

Table 1
Chemical compositions (wt.%) of the polycrystalline Ni (99.882%) used in the present study.

C	Si	Mn	P	S	Cr	Fe	Al	Co	Cu	Ti	Mg	Ni
0.003	0.009	0.003	0.003	<0.001	0.004	0.021	0.014	0.004	0.005	0.048	0.003	99.882