Applied Surface Science 280 (2013) 398–404

Contents lists available at SciVerse ScienceDirect

Applied Surface Science

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

X-ray diffraction study of residual elastic stress and microstructure of near-surface layers in nickel-titanium alloy irradiated with low-energy high-current electron beams

L.L. Meisner[a,b], A.I. Lotkov[a], M.G. Ostapenko[a,c,*], E.Yu. Gudimova[a]

[a] Institute of Strength Physics and Materials Science SB RAS, 2/4 Akademichesky Ave., Tomsk, 634021, Russia
[b] National Research Tomsk State University, 36 Lenin Ave., Tomsk, 634036, Russia
[c] National Research Tomsk Polytechnic University, 30 Lenin Ave., Tomsk, 634050, Russia

ARTICLE INFO

Article history:
Received 25 March 2013
Received in revised form 29 April 2013
Accepted 30 April 2013
Available online 20 May 2013

Keywords:
Nickel-titanium alloy
Electron beam treatment
Surface layers
X-ray diffraction analysis
Gradient microstructural changes
Residual elastic stresses

ABSTRACT

In the work, we compare quantitative estimates of residual stresses in nickel-titanium (NiTi) alloy surface layers after electron beam treatment. The quantitative estimates to be compared were taken using X-ray diffraction (XRD) techniques with symmetric and asymmetric Bragg diffraction geometries. A method of quantitative X-ray diffraction estimation of residual stresses in materials with gradient changes in microstructure and physical properties, including elastic moduli, is described. It is found that in a NiTi specimen with one side irradiated by a low-energy high-current electron beam, the maximum residual elastic stresses $\sigma \approx 550$ MPa are localized in the modified surface layer (melted by the electron beam and rapidly quenched), whereas the residual elastic stresses in the underlying layer with initial B2 structure are no greater than ~100 MPa. It is for this reason that stress-induced B19′ martensite is formed in the material layer beneath the modified layer.

1. Introduction

One of the factors largely responsible for many physicomechanical properties of metal materials is the presence of internal or residual elastic stresses in the materials after removal of external deforming forces. These stresses may arise, for example, due to the action of charged particle flows of varying nature: ions, electrons, plasma jets. The rise of internal stresses can be associated with structural phase transformations in and near the material zone directly affected by charged particle flows. In X-ray diffraction (XRD) methods, the internal stresses are determined through experimental measurements of the relative change in lattice spacings $\Delta d_{hkl}/d_{hkl}$ [1], which is called the lattice strain, and quantitative estimation of the stress tensor components by Hooke's law (assuming that the lattice strain is elastic) with regard for loading types [2]. However, it is not quite clear and still debatable to what extent XRD data reflect actual variations in internal stresses in deep material layers, what values of elastic constants are most acceptable for XRD estimation of stresses, and whether mechanically measured elastic moduli are applicable in similar estimation. These questions are particularly hard to answer where external action causes a gradient change in chemical composition and microstructure, which is characteristic of materials with surface layers modified by electron and ion plasma flows. Evidently, in this case, direct use of table elastic constants appropriate to a homogeneous material structure is not justified.

Among the now available techniques of residual stress estimation, X-ray diffraction methods are most informative for analysis of surface layers. These methods take into account not only variations in lattice strains but also in elastic constants in the presence of residual stresses. This issue is addressed in monographs [2–5], reviews [6–13], and in many original scientific papers, e.g. [14–24]. The most complete coverage of modern XRD methods has been given by Hauk and Vaessen [4,8,15], Genzel [9,16–18], and Welzel [5,10,20].

The problem of residual stress estimation in surface layers is of particular significance for NiTi-based alloys due to increasing interest in surface modification and thin coating deposition on these materials. Under certain conditions, the presence of residual stresses in surfaces layers of NiTi alloys can either provoke or suppress thermoelastic phase transformation from a high-temperature B2 phase to a martensite phase. Both effects produce considerable changes in the basic functional properties of the alloys: superelasticity or shape memory. Therefore, it is of importance to search for ways of accurate residual stress estimation in near-surface layers of NiTi-based materials after electron beam treatment.

* Corresponding author. Tel.: +7 89234089987; fax: +7 3822492576.
E-mail addresses: artifakt@ispms.tsc.ru, ostapenko_mg@sibmail.com (M.G. Ostapenko).

0169-4332/$ – see front matter © 2013 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.apsusc.2013.04.168

The aim of our work is to quantitatively estimate the residual stresses in surface layers of NiTi specimens after electron beam treatment with surface melting. For this purpose, we use a generalized method that combines different Bragg–Brentano geometries and takes into account gradient variations in elastic moduli.

2. Material and research techniques

The alloy under study was melted from iodide titanium and NO-grade nickel of equiatomic ratio in an electric arc furnace (with six-fold remelting of the ingot). The ingot was homogenized at T = 1273 K for 6 h and then cooled in the furnace. The test specimens of dimensions $15 \times 15 \times 1$ mm prepared from the ingot by electroerosion cutting were subjected to chemical surface cleaning, annealing at T = 1073 K for 1 h with subsequent cooling in the furnace, and electrolytic polishing. As a result, the initial NiTi specimens at room temperature were in the two-phase state: a main phase with B2 structure (bcc, CsCl ordering, start temperature of direct B2 → B19′ martensite transformation $M_S = 283K$, lattice parameter $a^0_{B2} = 3.0132 \pm 0.0005$ Å corresponding to $Ti_{49.5}Ni_{50.5}$) and a small Ti_2Ni phase amount (<5 vol. %).

Electron beam treatment was conducted at the Institute of High Current Electronics SB RAS (Tomsk, Russia). The NiTi specimens were subjected to pulsed (pulse duration $\tau = 150$ μs, number of pulses $N = 5$) surface irradiation by a low-energy ($U = 15$ kV) high-current ($I = 70$ A) electron beam of energy density $E = 20$ J/cm^2 with surface melting under the conditions of high vacuum (~10^{-6} Pa) with oilless pumping.

XRD analysis of structural phase states in NiTi near-surface layers after electron beam treatment was performed at room temperature on the equipment of the Shared Use Center "Nanotech" of ISPMS SB RAS (Tomsk, Russia): on a DRON-7 diffractometer (Burevestnik, Russia) in Co-K_α radiation (with a Fe-filter for cutoff of Co-K_β radiation) and on a Shimadzu XRD-6000 diffractometer (Shimadzu, Japan) in Cu-K_α radiation (with a monochromator for cutoff of Cu-K_β radiation). The phase composition and the structure of surface and deeper layers were analyzed using X-ray diffraction in symmetric Bragg diffraction geometry (θ–2θ mode) and grazing incidence X-ray diffraction in asymmetric Bragg diffraction geometry with a varying incidence angle α, which is the angle between the specimen surface plane and the direction of a primary incident X-ray beam (Fig. 1). The imaging conditions and the choice of X-ray wavelengths, K_β-filters, and glancing incidence angles α for NiTi specimens were described by us earlier [25,26]. Here we consider that the data obtained from diffraction patterns in symmetric Bragg diffraction geometry characterize the specimen structure in the material bulk, and those obtained from diffraction patterns in asymmetric Bragg diffraction geometry characterize the structure of a layer whose thickness corresponds to a chosen glancing incidence angle α. X-ray patterns in asymmetric Bragg diffraction geometry were taken at glancing incidence angles α = 3°, 6°, and 12°.

The thickness h of the analyzed NiTi layer was estimated by the formula [27]:

$$h = -\ln(1 - R)/\mu k \quad (1)$$

where R is the radiation absorbed by a layer of thickness h, μ is the linear absorption coefficient calculated for NiTi alloys, $k = (1/\sin\alpha + 1/\sin\delta)$ is a coefficient that takes into account diffraction geometry, $\delta = (2\theta - \alpha)$ is the angle between the X-ray beam reflected from the plane (hkl) and the specimen plane, θ is the Bragg angle of a family of lattice planes (hkl).

The lattice parameters of the B2 phase of NiTi was determined according to [28] with construction of extrapolation dependences of the lattice parameter of the examined phase on the function $f(\theta) = \frac{1}{2}\left(\frac{\cos^2\theta}{\sin\theta} + \frac{\cos^2\theta}{\theta}\right)$ at a measurement accuracy $\Delta a = \pm 0.0005$ Å.

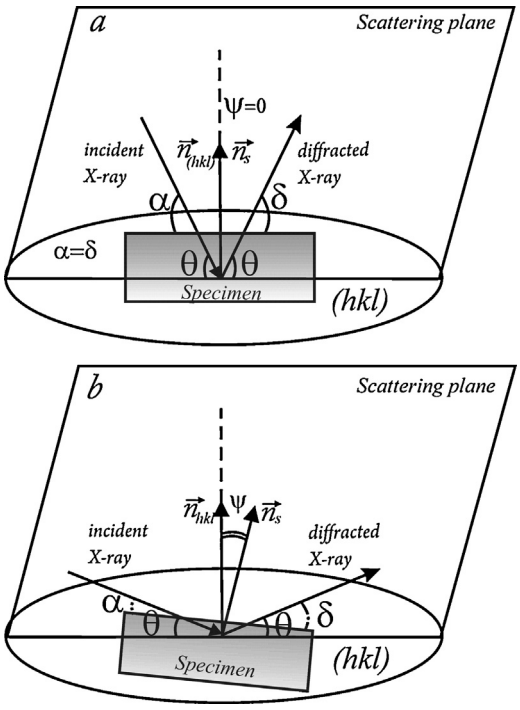

Fig. 1. Schematic diagrams of the specimen position, incident X-ray beam, and diffracted X-ray beam in symmetric (a) and asymmetric Bragg diffraction geometries (b); $\vec{n_S}$ and $\vec{n_{hkl}}$ are the normal vectors to the surface and reflection planes, respectively.

With the aim to estimate the residual stresses in the irradiated NiTi specimens and to compare the estimates in reliability and applicability to study heterogeneous structures with gradient properties and lattice strains, three XRD methods were used. *Method 1* was a standard "$\sin^2\psi$" method [29]. *Method 2* was developed by us earlier [25] and was used to study materials with modified surface layers or coatings; this method describes the conditions under which asymmetric Bragg diffraction geometries can be used for analysis of residual stresses by the "$\sin^2\psi$" method. *Method 3*, being based on estimation of residual stresses in materials with gradient structures, allowed taking into account gradient variations in both lattice strains and elastic moduli in residual elastic stress fields through a combination of *method 2* and the ways proposed by Hauk and Vaessen [4,8,15], Genzel [9,16,18], and Welzel [10,20]. More detailed description of *method 3* is given below.

The residual elastic stresses $\boldsymbol{\sigma}$ in metal specimens are impossible to directly measure; these stresses can only be calculated from strains $\boldsymbol{\varepsilon}$ using equations of elasticity theory. The elasticity theory assumes that the applied load is not too high, the strain is elastic, and the deformed solid regains its initial sizes after unloading. In XRD methods, the strains are determined from variation in lattice spacings d_{hkl} in a stressed crystallite. In the general case, the elastic stress and the strain are related by Hooke's law [2,30]:

$$\sigma = E\varepsilon \quad (2)$$

where E is the elastic modulus, ε is the lattice strain in a phase examined. The stress σ characterizes the bulk stress state of a specimen and can be represented by expansion in terms of three principal normal stresses σ_1, σ_2, and σ_3 in the directions of principal strain axes (Fig. 2) [11,24]. Each element in the volume of a stressed specimen experiences three principal stresses, whereas in its near-surface region, to which XRD methods are limited, $\sigma_3 = 0$