The Reverse Indentation Size Effect in Heavily Deformed Materials

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1. INTRODUCTION

Experiments performed on a large number of coarse-grained (CG) materials have shown that the value of the indentation load P (the depth of indenter penetration h), as a rule, can have a significant effect on the measured value of microhardness H_V. This effect is called the "indentation size effect" (ISE). It is usually observed in the region of low loads and consists in an increase in microhardness as the indentation load decreases **(Fig. 1, *a*, *curve 3*)**. The effect has a different nature, but most often two reasons lead to it: the introduction of geometrically necessary dislocations (GNDs) during the indentation of crystalline materials that ensures the formation of an indent and the violation of the homogeneity of the surface layer caused by mechanical treatment of the sample surface before indentation.

The aim of this work was to study the indentation load effect on the microhardness of materials subjected to severe plastic deformation (SPD). The dependences $H_V(P)$ obtained on the number of ultrafine-grained (UFG) and nanocrystalline (NC) materials, namely, VT1-0 [1], Al [2], Cu [3] and Al-Li [4], subjected to cryorolling, equal channel angular pressing, direct hydroextrusion or combined direct and equal channel angular hydroextrusion were analyzed.

2. ISE in CG and UFG&NC SAMPLES

It is shown that in CG samples of all materials studied the usual ISE is observed: the microhardness value increases with decreasing the indenter load (indenter penetration depth); the dependences can be described within the framework of a simple GNDs model developed by Nix and Gao [5]. According to this model the relation between the microhardness H and the penetration depth of the indenter h in the case of a structurally homogeneous material is described by the following expression:

$$\frac{H}{H_0} = \sqrt{1 + \frac{h^*}{h}},$$

where H_0 is the hardness of the material in the absence of the geometrically necessary dislocations, for instance for $h \gg h^*$ (the "true" or classical hardness), h^* is the characteristic length.

Fig. 1,*b* shows that this expression describes the dependence of microhardness on the depth of indenter penetration for samples of CG copper (curve 3), UFG Cu (curve 2) at $h > 4$ μm and UFG Cu (curve 3) at $h > 2$ μm [3]. The same can be seen in **Fig. 2** at indentation depth $h > 1$ μm for samples of VT1-0 titanium [1].

The model [5] predicts that the microhardness of a rigid material should not strongly depend on the depth of indenter penetration. This is actually observed in measurements the microhardness of the samples preliminarily hardened by different SPD methods. An increase of microhardness with decrease of the load was very small or practically not registered during the indentation of VT1-0 [1], Al [2], Cu [3] and Al-Li [4]. This can be seen in **Fig. 1,*a* curves 1 and 2, and Fig. 2, curves 2 and 3**.

3. REVERSE ISE

Of some interest is the significant drop in the microhardness of SPD processed specimens in the region of low loads as can be clearly seen in **Fig.1,*a*, curves 1 and 2** (**reverse ISE**). This effect may be due to the surface treatment (grinding and polishing) of the sample prior to indentation, which led to a violation of the structural homogeneity of the surface layer.

It is known that grinding usually hardens the surface layer of cast and annealed specimens. Pretreatment of the material by various SPD methods leads to the formation of a special structure in its volume that provides high microhardness. In this case, additional grinding of the surface destroys to some depth the microstructure created during SPD and thereby softens the surface layer. Experimentally this manifests itself in a decrease of the measured microhardness at low loads, which was observed in SPD processed materials Al [2], Cu [3] **(Fig. 1,*a*),** Al-Li [4].

The dependence $H_V^2\left(\frac{1}{h}\right)$ allowes to estimate the thickness of the disturbed layer as shown in **Fig. 1,*b*** and **Fig. 2***;* it is large for soft material, such as Al or Cu, and noticeably less for stiff one, such as titanium.

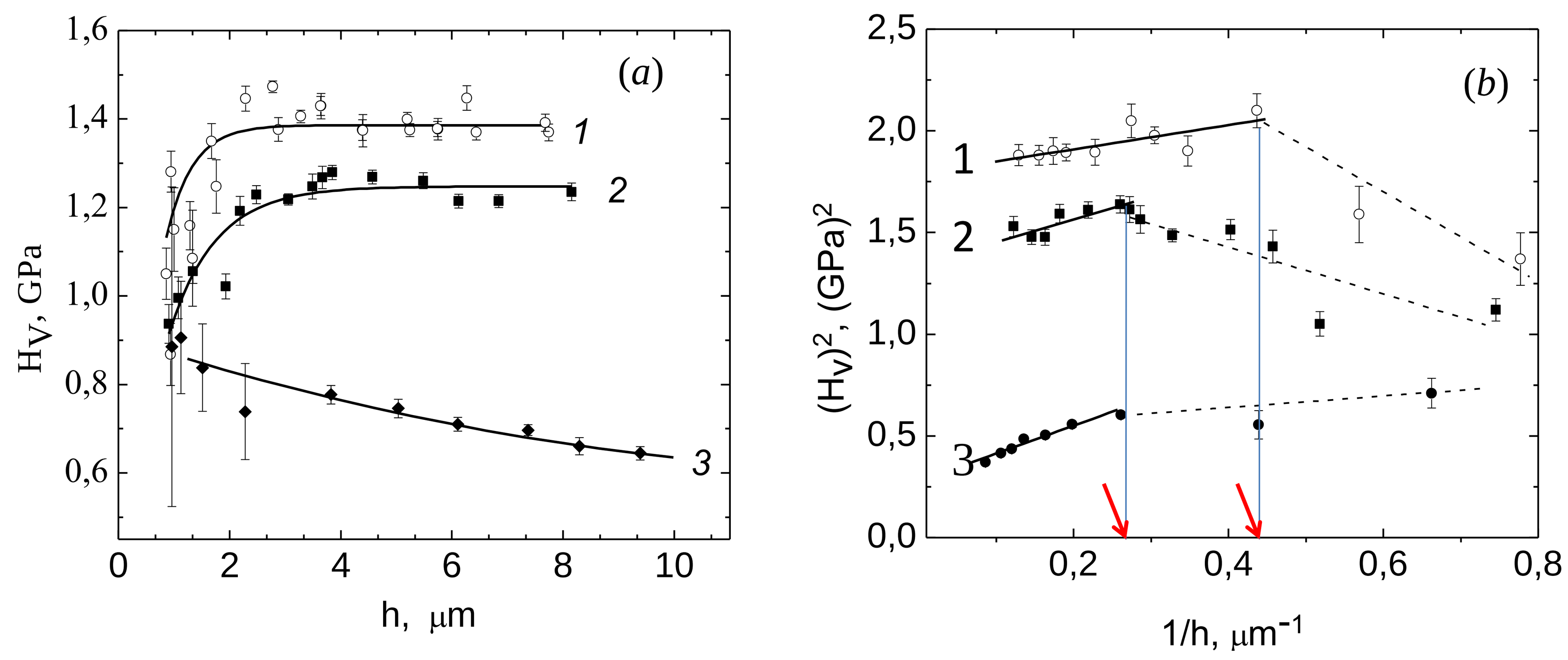

Fig. 1. a) - dependences of microhardness of **Cu** on the penetration depth of the indenter in samples with different grain sizes: 1 – 0.5 μm, 2 – 0.6 μm, 3 – (1-10) μm [3]; b) $H_V^2\left(\frac{1}{h}\right)$ dependences for the same samples according to the expression of Nix and Gao [5].

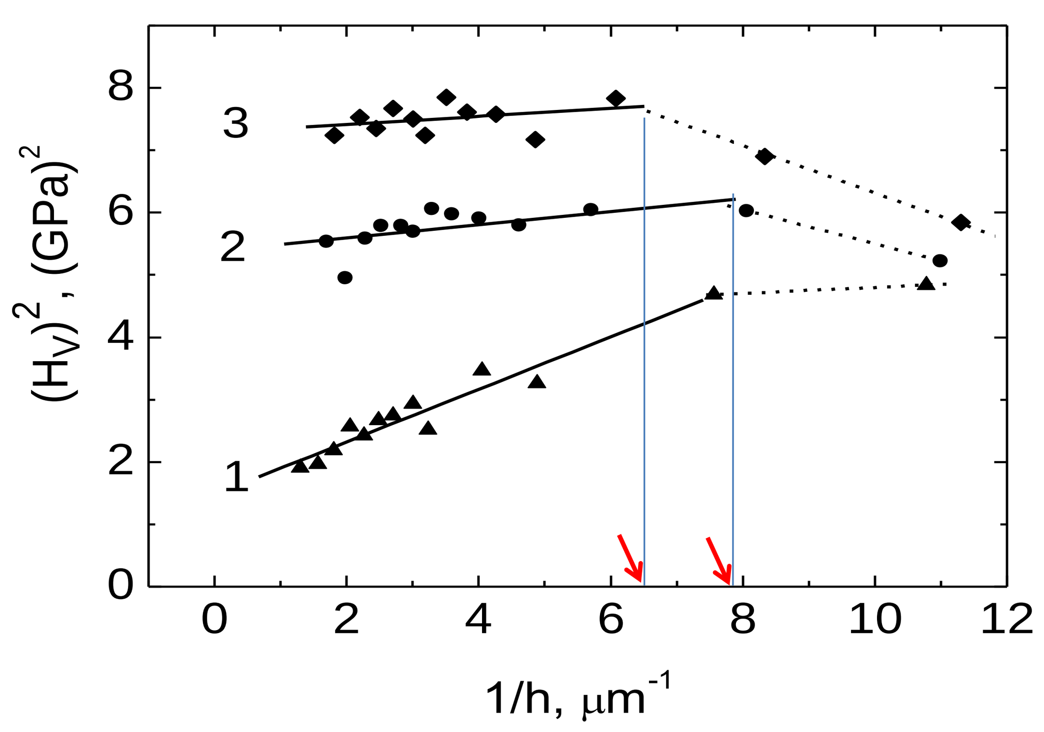

Fig. 2. Dependence of the microhardness of **VT1-0** titanium samples on the penetration depth of the indentor: as-supplied and rolled to strains e = 0.6 (2) and e =1.3 (3). [1] $e = \ln(t/t_0)$ is the true deformation upon cryorolling, t_0 and t are the initial and final thickness of the rolled plate, respectively.

The arrows in **Fig. 1,*b*** and **Fig. 2** indicate the depth of penetration of the indenter, at which (and at greater depths) the measured microhardness characterizes the property of a homogeneous strengthened material. Consequently, the thickness of the layer damaged by grinding in SPD-hardened copper is approximately (2-4) μm, and in titanium it is significantly less than 1 μm.

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