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Letter

Mechanical properties of V_2O_5 polycrystals grown by laser light irradiation

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Abstract

Microhardness, stiffness and elasticity parameters of V_2O_5 polycrystals grown by irradiation with continuous wave IR laser light (IR ND:(Y–Al–garnet)) are discussed. Structural and metallographic analysis showed that the mechanical parameters strongly depend on the lateral and vertical directions with respect to the area irradiated. These dependences were attributed to the structural changes in polycrystals grown as a result of standing heat waves formed due to irradiation by laser light.

1. Introduction

It has been shown earlier that, under the influence of IR continuous wave (CW) laser light irradiation in an oxidizing atmosphere (*e.g.* air), different oxide structures on metallic surfaces could be easily grown [1–4].

The mechanical, optical and electrical properties of samples prepared by isothermal (*e.g.* furnace) and laser methods are markedly different [5]. In particular in a previous paper it has been shown that the microhardness of oxide layers is two or three times higher than for V_2O_5 single crystals grown from melts by slow cooling [6]. In the present letter we describe the results of our investigations concerning the microhardness, stiffness and elastic constants of samples irradiated by IR laser light.

2. Experimental details

The samples of chemically cleaned metallic sheets of vanadium of size $30 \times 5 \times 0.3$ mm^3 were irradiated with IR CW Y–Al–garnet laser

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($\lambda = 1.06$ μm, $P = 10$–30 W, $\tau = 10$–100 s) at a 90° angle of incidence, focussing the beam in a circular spot of 3 mm radius. The kinetics of the oxidation process was followed by the method described in ref. 6.

The oxides formed on the vanadium surfaces were examined by optical microscopy (Olympus BHT-2) and in more detail by scanning electron microscopy techniques (Philips 515). The oxygen:vanadium ratio was analysed by microprobe techniques (JEOL-733). Microhardness measurements were carried out with a Leitz Miniload II tester along the length of the sample surface at intervals of 0.5 mm and with a nanoindenter (Nano Instruments, TN) in the vertical direction through the thickness of the oxide layer. The computational methods have been reported earlier [7, 8].

3. Results and discussion

Typical micrographs of the oxides grown have been presented in ref. 1. The wave-like structure of different oxides was clearly seen. Scanning electron micrographs taken from the different areas of the sample show different mosaic-like patterns of V_2O_5, V_2O_3 and VO_2 oxides. The types of oxide were identified by colour test and microprobe techniques. From these figures we can conclude that the thickness and compounds of oxides formed are different along the sample. Taking into account that the oxide growth rate strongly depends on the temperature and the oxide:metal ratio, from

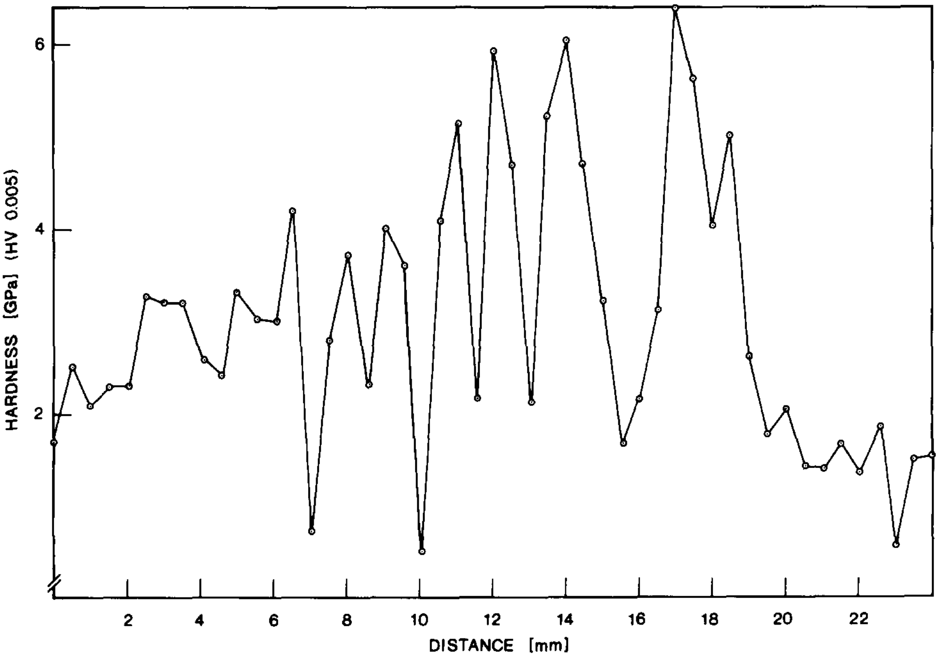

Fig. 1. Microhardness *vs.* distance measured on the surface of the irradiated sample.

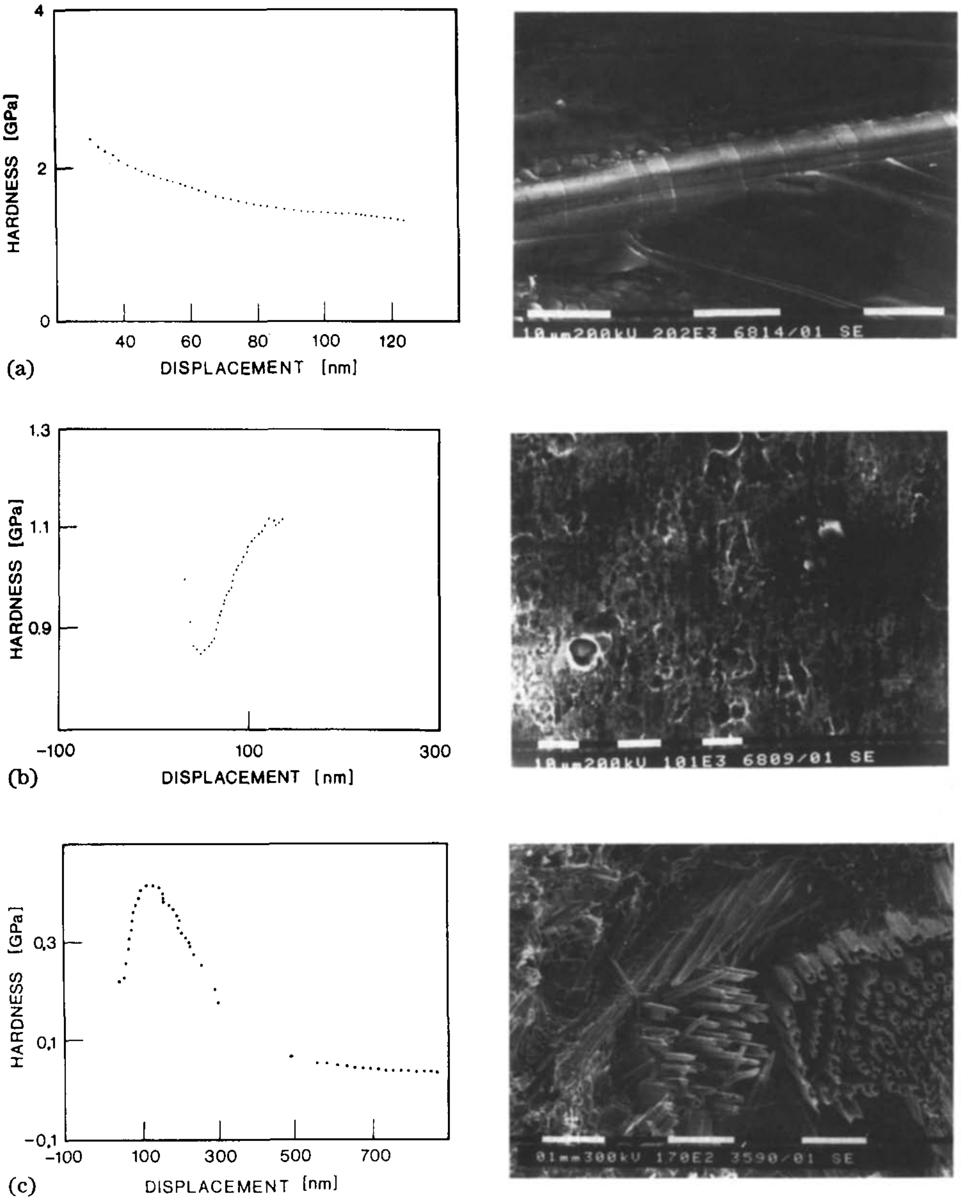

Fig. 2. (a) Microhardness change in the depth and the morphology of the area far from the zone of irradiation. (b) Microhardness change in the depth and the morphology of the area in the vicinity of the laser spot. (c) Microhardness change in the depth and the morphology of the area inside the laser spot.

the gradient of temperature we can see that the oxide formation could be attributed to the standing-wave-like heat distribution established on the sample surface during the irradiation process. The standing heat wave structure is a result of interference of heat waves coming from the laser spot to the non-irradiated area of the sample and reflected from boundaries (edges of the sample). Direct temperature measurements carried out using thermocouples imbedded into the back of the sample have confirmed this assumption.

The results of microhardness measurements show the same wave-like distribution (Fig. 1). From a mathematical analysis it was derived that two types of microhardness oscillation could be distinguished: oscillation with wavelength $\lambda = 2.5$ mm at the laser spot and in its vicinity; and oscillation with wavelength $\lambda = 3.5$ mm far from the laser spot.

We note that microhardness measurements could be made everywhere on the sample except in the top area of the irradiated part (from 0.8 mm and beyond). The thickness of oxide layer at the central part of the irradiation was about 0.3–0.7 mm while in other places it was only 0.05–500 μm. It should be noted that in the "heights" (in the top part of the thickest oxide layer) we could observe mostly V_2O_5 and V_2O_3 structures while in long valleys (far from the laser spot) we observed VO_2. This could be connected with different values of the rate of temperature change with distance dT/dx at different locations along the sample.

The hardness measurements in the depth throughout the different oxide structural layers showed different values from one area to another. Results obtained from the area far (8 mm) from the spot of irradiation (Fig. 2(a)) revealed the hardness value changing from 2.3 GPa at the surface to 1.3 GPa at the bottom near the metallic matrix. Calculations based on nanoindenter measurements showed that the stiffness of this part of the layer is about $G = 1.69 \times 10^5$ N m^{-1} and the elastic modulus is about $E = 1.237 \times 10^2$ GPa. The thickness of the oxide layer at this point was 120 nm.

Measurements made 5 mm from the laser spot (Fig. 2(b)) show a softer oxide layer of hardness 0.9 GPa at the upper surface, which is even softer at a depth of 50 nm. An abrupt increase in hardness up to 1.12 GPa at 140 nm depth was measured. The thin oxide layer showed less stiffness (4.83×10^4 N m^{-1}) and had a lower modulus of elasticity (0.43×10^2 GPa) than the layer shown in Fig. 2(a).

Measurements made near the laser spot (1 mm from the centre) were very difficult because of the rough nature of this area and the existence of long microcrystals of V_2O_5 (Fig. 2(c)). However, some data were obtained with respect to the layer thickness (1 μm) and hardness values of 0.05–0.45 GPa were measured throughout the oxide layer.

4. Conclusion

The oxide formation on the metallic surface under the influence of CW laser light could be linked with the formation of standing heat waves leading to oxide structures with different microhardnesses, stiffnesses and elasticities due to the dT/dx variation. Vickers hardness measurements showed that the oxides are softer at the centre (rich in V_2O_5). The developed mosaic-like structures (almost single crystals) are softer than polycrystals. Non-linearities observed in the microhardness variation along the sample length were attributed to inhomogeneous standing-wave-shaped heat profiles during oxide formation. The appropriate choice of sample geometry and irradiation conditions might lead to the formation of oxide layers with specific values which might be of importance in some applications.

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