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Optical technique of investigation of the structural changes of aluminum polycrystals

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An *in situ* technique has been proposed to observe structural changes of an aluminum polycrystal during plastic straining based on white light scattering by the previously etched sample surface. It is shown that a quasi-periodic relief of grain surfaces results from the chemical etching, the character of such relief is defined by crystallographic orientation of each grain. The white light scattering by the surface relief causes coloring of grains which is recorded using a Web-camera. It is shown in experiment that a change in crystallographic orientation of grains or their fragments during the plastic straining of two-dimensional aluminum polycrystals is accompanied by coloring change thereof.

Предложена методика наблюдения "*in situ*" структурных изменений в процессе пластической деформации двумерных поликристаллов алюминия, которая основана на использовании рассеяния белого света поверхностью предварительно протравленного образца. Установлено, что в результате химического травления на поверхности каждого зерна формируется квазипериодический рельеф, характер которого определяется кристаллографической ориентацией зерна. Рассеяние белого света на поверхностном рельефе зерен приводит к их цветовому окрашиванию, которое регистрируется WEB-камерой. Экспериментально установлено, что изменение кристаллографической ориентации зерен или их фрагментов в процессе пластической деформации двумерных поликристаллов алюминия сопровождается изменением их цвета.

It is well-known that the metallographic detection technique of polycrystal structure consists as a rule in the chemical etching. To reveal the grain boundaries in aluminum, the Keller etchant [1] is used most often. It is possible to detect using chemical etching not only grain boundaries as thin dark lines but also an anisotropic quasi-periodic relief as the etching grooves on the surfaces of grains, the character of such relief being defined by crystallographic orientation of each grain. Interaction of white light with this relief at a certain selected light source and positional relationship of the sample, the light source and the recording device may result in coloration of grains depending on their orientation. During a plastic straining, this experimentally ascertained fact allows to observe *in situ* not only changes in a grain orientation, but also orientation changes within each grain simultaneously for the whole sample.

Recently, the major attention is directed towards the coloration problem of various sample surface parts in photomicrograph depending on the orientation factor and other parameters (for example, the scattering factor in [2]). According to [3], it is possible to see various grain orientations in polycrystalline aluminum using so-called orientation color maps. In [4], orientation color maps are presented for steel samples in as-prepared and strained states. In [5], the color map makes it possible to judge the local strains in various areas of copper polycrystals after plastic straining. In [6], the orientation maps illustrate evolution of a microstructure in pure titanium after straining. It is to note that all orientation color maps in [2–6] are obtained artificially.

Fig. 1. Color orientation map of an aluminum polycrystal surface (×2), the whole sample.

As a rule, the orientation of individual areas of a sample is determined by diffraction methods, and orientation color maps are obtained using special software which colors sample map in accordance with orientation data (or other data). Thus, to construct an orientation color map, it is necessary to obtain orientation data for various parts of a sample.

The method described in this work allows to obtain automatically the color orientation maps for samples of about 100×20 mm^2 size. The sample is illuminated by white light from two sources: a filament lamp and a luminescent lamp. The first light source is placed near the sample (at 20 cm distance) while the second source and the sample are 200 cm distant. The light scattered by the sample surface is recorded using a PC-CAM 300 Web-camera connected with a computer. Between a sample and the Web-camera the system of lenses is used to provide a blowup. Fig. 1 presents the color orientation map of aluminum polycrystal sample after chemical etching using Keller etchant [1] during 15 s. As is shown below, the observed color of each grain is defined by its crystallographic orientation. It is purpose of this work to explain the physical nature of the grain coloring. The aluminum sample fragment containing 5 grains was selected to that end. The color orientation map of this fragment surface obtained using a Web-camera is shown in Fig. 2. The grains differing in crystallographic orientation have different colors. Fig. 3 shows the grain orientation using a stereographic triangle. The grain surface photomicrographs obtained using a scanning electron microscope JEOL JSM-840 are shown in Fig. 4, too. A quasi-periodic relief is observed on the surface of each grain. This relief causes the light scattering anisotropy. To test the validity of this explanation, an additional experiment has been realized using monochromatic radiation of the He–Ne laser (λ = 633 nm, power $P = 2$ mW). The sample fragment containing 5 grains was placed at the holder which can be moved in two directions transversely to laser beam. Thus, it was possible to direct the laser beam successively to the surface of each grain. The light spot diameter at the sample surface was 1 mm. Fig. 3 shows the character of the laser beam scattering by surface of each grain. The anisotropic pattern of monochromatic radiation scattering correlates with the character of the grain surface relief. For surface relief which consist only one set of the etching grooves, the uniaxial-anisotropic pattern of the laser beam scattering in the direction transverse to these grooves is observed. If the grain surface comprises two sets of the etching grooves, the laser beam scattering anisotropy is characterized by two directions. Thus, the grain surface relief arising from the chemical etching causes anisotropic light scattering. If the whole sample surface is illuminated by white light from two sources, the white light scattering patterns at the quasi-periodic relief of the grain surfaces can be observed using digital camera (Fig. 1). Thus, the results of the last experiment verify the possibility of the polycrystal grains coloration which is due to the interaction of white light with

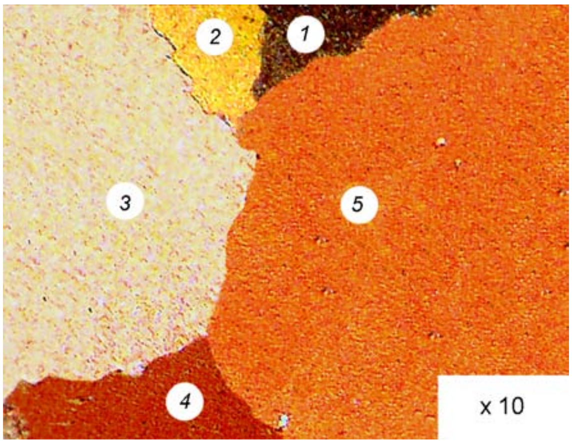

Fig. 2. Color orientation map for a fragment of aluminum polycrystal surface (×10).

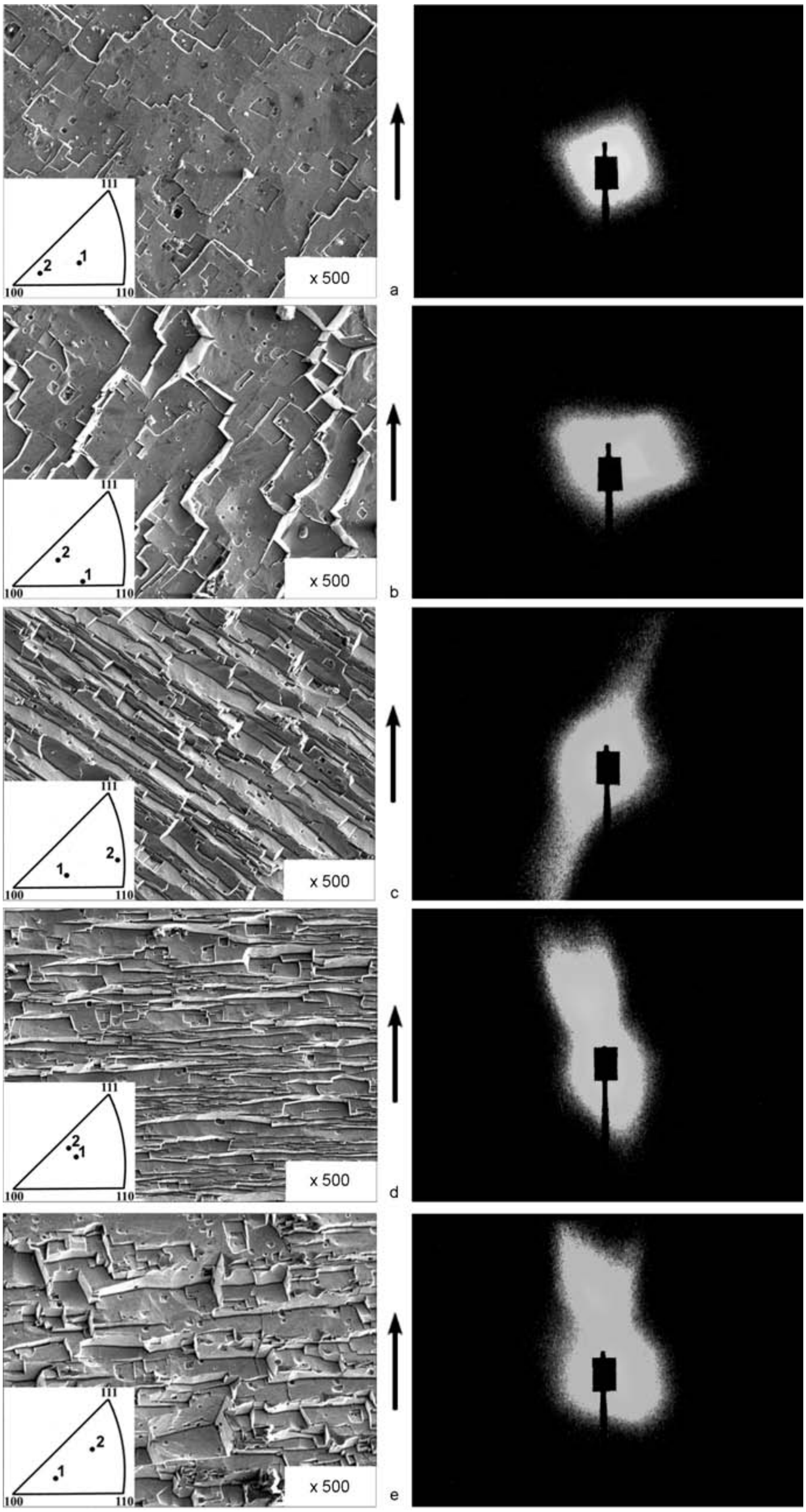

Fig. 3. The grain surface scanning electrons photomicrographs (×500), laser beam scattering patterns by grain surfaces, crystallographic orientation of grains (1 — normal direction, 2 — direction shown by arrow): grain 1 (a), grain 2 (b), grain 3 (c), grain 4 (d), grain 5 (e).

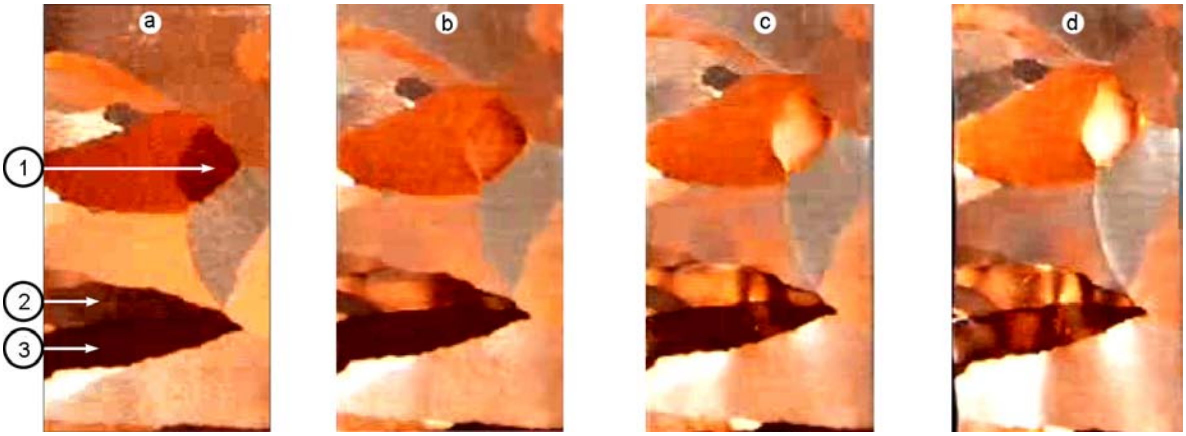

Fig. 4. Color orientation maps for two-dimensional aluminum polycrystal surface obtained in situ during plastic straining (1 — rotation of grain as a whole, 2, 3 — fragmentation of grains): $\varepsilon = 0$ (a), $\varepsilon = 12.6$ % (b), $\varepsilon = 17.9$ % (c), $\varepsilon = 23.3$ % (d).

the quasi-periodic relief of the grain surfaces. If the same positional relationship of a light source, a recording device and a sample are provided during a plastic straining, it is possible to observe in situ changes in crystallographic orientation of individual fragments simultaneously to the whole polycrystalline sample as color change of these fragments. Orientation maps of the aluminum polycrystal for various straining extents are shown in Fig. 4. The sample was strained under active tensile conditions at a constant straining rate $\dot{\varepsilon} = 1.2{\cdot}10^{-5}$ s^{-1}. The Web-camera used allows to obtain color orientation maps for a sample surface at a frequency of 30 pictures per second. Thus, the mechanism of various rotational structures rise and development and other orientational effects which define the sample plasticity can be traced. In particular, for the first time, the rotation of a grain as a whole (Fig. 4) during a plastic straining of two-dimensional aluminum polycrystal has been observed using the optical technique described above. As shown in Fig. 4, the color of the grain 1 changes continuously, while color of neighboring grains remains almost unchanged. In another area of a sample (grains 2, 3), the fragmentation of grains is seen which arises at the strain $\varepsilon = 12$ % and develops up to the sample failure at $\varepsilon = 23.3$ %.

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Оптичний метод дослідження структурних змін у полікристалах алюмінію

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Запропоновано методику дослідження "*in situ*" структурних змін у процесі пластичної деформації двовимірних полікристалів алюмінію, яка грунтується на використанні розсіювання білого світла поверхнею попередньо протравленого зразка. Встановлено, що у результаті хімічного травлення на поверхні кожного зерна формується квазіперіодичний рельєф, характер якого визначається кристалографічною орієнтацією зерна. Розсіювання білого світла на поверхневому рельєфі зерен приводить до їх кольорового фарбування, яке спостерігається за допомогою WEB-камери. Експериментально встановлено, що змінення кристалографічної орієнтації зерен або їх фрагментів у процесі пластичної деформації двовимірних полікристалів алюмінію супроводжується зміною їх кольору.