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New approach to grain boundaries detection in polycrystalline materials by scratching of sample's surface

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It was experimentally developed the new approach of the grain boundaries detection in the polycrystalline samples. In this approach scratches are applied on polished surface using the method of sclerometry, then the sample surface is illuminated by the light beam directed toward the surface at a small angle perpendicular to the direction of the applied scratches. The proposed approach is based on the anisotropy of the mechanical properties of the crystal sample. It is determined that the configuration of the pile-up of material which extruded on the grain surface as a result of the applying the scratches depends on the crystallographic orientation of the grain. Optical microscope image of the configuration of the pile-up in the area of scratch under oblique illumination of the sample surface considerably changes under crossing of the grain boundary. These changes allow clearly record the grain boundary.

Экспериментально получен новый способ выявления границ зерен в поликристаллических образцах. Способ заключается в нанесении на полированную поверхность царапин методом склерометрии и освещении поверхности образца пучком света под малым углом к ней перпендикулярно направлению нанесения царапин. Метод основан на анизотропии механических свойств кристаллического образца. Установлено, что профиль материала образца, вытесненного на поверхность зерна при нанесении царапины, зависит от кристаллографической ориентации зерна. Изображение в оптическом микроскопе профиля материала в области царапины при косом освещении поверхности образца резко меняется при пересечении границы зерен и это изменение позволяет точно определить положение границ зерен.

1. Introduction

The sclerometry is a well-known method for determining of the mechanical properties of materials, such as material hardness [1, 2]. In this method the indenter after penetration in the sample, is shifted along the sample's surface. This forms a scratch (groove) in such a way that the material under indentation zone reproduces the shape of the indenter with pyramid geometry. Other part of material is extruded on the surface of the sample toward the edge of the groove. The hardness of the material can be determined by measuring of the groove's width.

The anisotropy of mechanical properties in the surface of material can be also studied with the sclerometry method. For this purpose scratches are applied in different directions. For example, the method of sclerometry with the use of Vickers diamond pyramid indenter with the apex angle of 136° was utilized in [3] during the investigation of the mechanical hardness of C_{60} fullerite single-crystal. It was shown experimentally that width of the groove depends on the crystallographic plane and the direc-

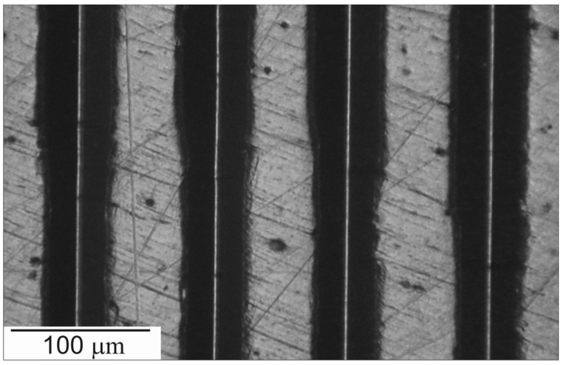

Fig. 1. Optical microscope image of the surface of bicrystal aluminum obtained under illumination by light beam directed perpendicularly to the surface after scratching the polished sample surface (scratches intersect the grain boundaries). The image was obtained by means of a PMT-3 device using Vickers diamond pyramid with a load of $P = 10$ g.

tion of scratching. Thus, if scratch is made on the surface of polycrystal, the width of the scratch should change in the areas of the intersections with the grain boundaries. This effect could provide a reliable mechanism to found out the location of the grain boundaries.

2. Experimental procedures

The aluminum and copper polycrystalline samples were used in this study. Fig. 1 shows the optical microscope image of the surface of the polished aluminum bicrystal in the area of the intersection of the grain boundaries with surface grooves. The grooves are formed by scratching of the surface of the bicrystal using Vickers diamond pyramid of the PMT-3 device with a load of $P = 10$ g. A distance between the scratches was 100 μm. All scratches intersect the grain boundary. Fig. 2 shows the optical microscope image of the same surface area on the reverse side of the sample after revealing of grain boundary using a Keller etchant [4]. In the intersections of scratch with the grain boundaries, the width of the grooves changes (Fig. 1), but this alteration is insignificant and can not be used as a reliable criterion for determination of the location of the grain boundary (Fig. 2).

The configuration of the "pile-up" in the area of the grooves which arises from scratching was examined using a Michelson-Linnik interferometer and optical microscope. This configuration of the "pile-up" can be schematically represented (Fig. 3) as a configuration of the groove (*A*) which reproduces the shape of the indenter (pyramid) and the configuration of the material (*B*) extruded on the sample surface at the edge of the groove.

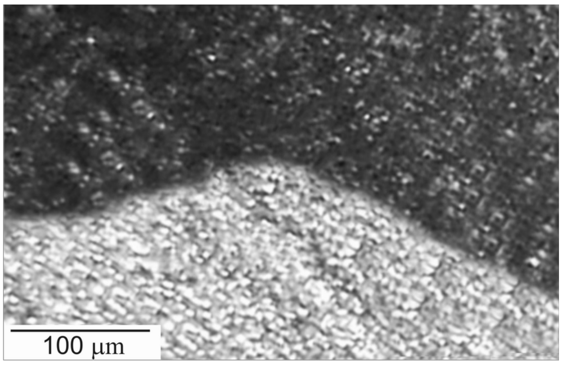

Fig. 2. Optical microscope image of the surface of bicrystal aluminum after the chemical etching of the reverse side of the sample with the purpose of the grain boundaries detection.

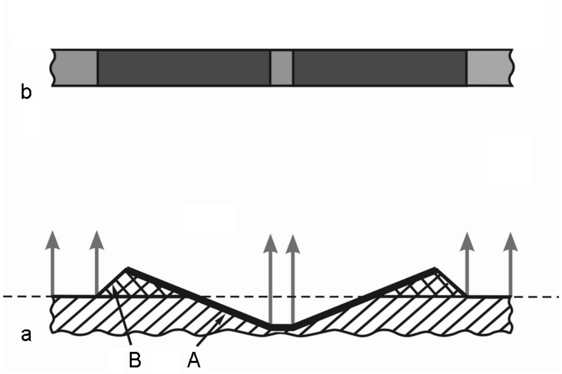

Fig. 3. Scheme of the image groove formation in the field of view of optical microscope under illumination by light beam directed perpendicularly to the sample surface: a) scheme of the configuration of pile-up in the area of groove; b) an image of the groove.

3. Results and discussion

Experimental investigations of the present work show that the configuration of the "pile-up" formed on material extruded on the grain surface, as well as depth and width of the groove depend on the crystallographic orientation of the plane of scratch applied and on the direction of the scratching. This dependence can be used to study the mechanical characteristics and, as it is shown below, to determine the location of the grain boundary in polycrystalline sample.

The groove image under the normal (standard for PMT-3 device) incidence of light on the sample surface is schematically presented in Fig. 3. It represents a white stripe in the central part of the groove image. It occurs as a result of the light

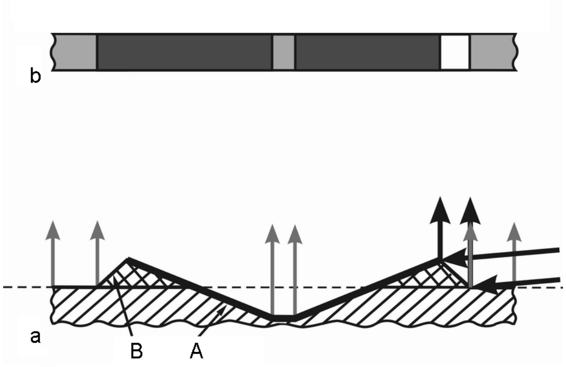

Fig. 4. Scheme of the image groove formation in the field of view of optical microscope under illumination by light beam directed perpendicularly to the sample surface and the additional oblique illumination (from the right) of the sample surface at the angle of ≈ 10° to the surface and perpendicularly to the direction of the scratches: a) scheme of the configuration of pile-up in the area of groove; b) an image of the groove.

reflection from indentation impression produced by the top of the four-sided Vickers pyramid, because four faces can not be reduced to the point [5]. This crntral part corresponding to the pressed-in section is almost parallel to the surface of the sample. Also, black stripes located left and right to the zone of the white stripe can be observed due to the rays reflected from the groove area which reproduces the shape of the pyramid indenter and it did not appear in the objective of optical microscope. The white stripes located on left and right sides of the area of the black stripe are formed as a result of the light reflection from the sample surface without scratches.

Additional oblique light beam directed at a small angle towards the sample surface on the area of the groove can provide the light reflection of the sample material extruded on the surface (Fig. 4). The corresponding groove image is schematically presented in Fig. 4. White strip should appear at the edge of the one of black stripes that is closer to the source of the oblique light. The white stripe arises as a result of light reflection from the part of the material which is extruded on the sample surface in the process of scratching. It is shown experimentally that the additional source of illumination must differ from the primary source of illumination by its intensity or type. Subsequently, the image of this white strip does not merge with the image of the polished surface without scratching.

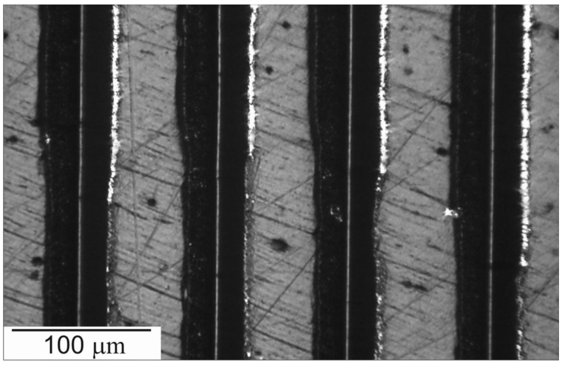

Fig. 5. Optical microscope image of the surface of bicrystal aluminum (the same fragment of the surface of bicrystal aluminum as shown on the Fig. 1) obtained under illumination by the light beam directed perpendicularly to the surface and the additional oblique illumination (from the right) of the sample surface at the angle of ≈ 10° to the surface and perpendicularly to the direction of the scratches.

The intensity of the light reflected from the material extruded on the sample surface is depended on the configuration of the "pile-up" of this material and its orientation with respect to the additional oblique light beam. The grooves are formed due to plastic deformation which is anisotropic. Thus, the configuration of the "pile-up" is determined by the crystallographic orientation of the scratch's plane as well as the direction of the scratching. The "pile-up" configuration alters in the places of the scratch intersection with grain boundaries. It may be possible to determine the location of the grain boundaries by visualization of the effect of anisotropy of mechanical properties. Indeed, the intensity of light reflected from the "pile-up" must depend on the crystallographic orientation of the scratch plane and the direction of the scratching, and should change in the places where the scratch intersects the grain boundaries.

Experimental confirmation of the possibility of this effect using is shown in Fig. 5. The figure presents the image of the same grooves as in Fig. 1, but obtained with help of additional oblique illumination of the sample surface at the angle of ≈ 10°. It can be seen that the white strip emerges in one of the grains on the edge of the black strip which is the closest to the additional source of oblique light. This white stripe practically disappears at transition to adjacent grain. Fig. 6 and Fig. 7 show the effect of the grain boundaries and twin boundaries

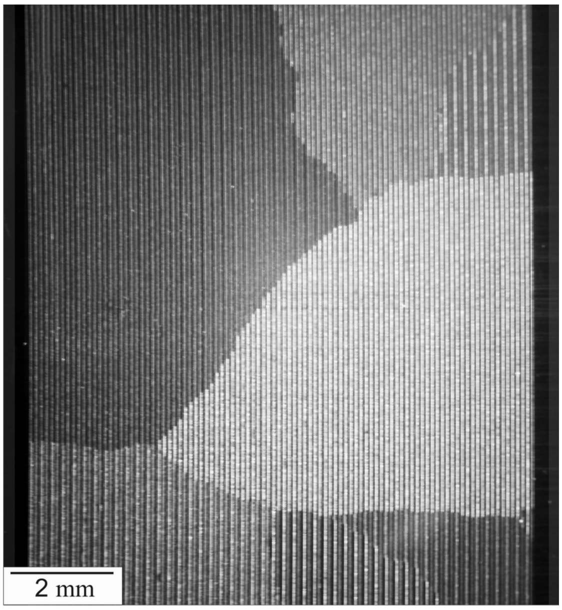

Fig. 6. Polycrystalline aluminum with identified grain boundaries after scratching the investigated polished surface by Vickers diamond pyramid indenter and additional oblique illumination. The scratches were applied with a normal load of 10 g with the distance between the scratches of 100 μm. Additional oblique illumination of the sample surface was applied at the angle of ≈ 10°.

detection in the polycrystalline aluminum and copper after applying scratches on a polished surface using the method of sclerometry and under illumination of the sample surface by the light beam directed in a small angle to the surface and normally to the direction of the scratches applied.

4. Conclusions

In the study it was experimentally developed the new approach to the grain boundaries and twin boundaries detection in the aluminum and copper polycrystalline samples using scratching their polished surfaces and illumination of the samples surface by the light beam directed toward a small angle to the surface and perpendicularly to the direction of the applied scratches. Differences in the configurations of the "pile-ups" formed by the extruded material and observed in the different grains or in the one grain but in different directions of scratching, are determined by the anisotropy of mechanical properties of the crystalline sample. Visualization of the effect of anisotropy of mechanical properties allows us to determine the location of the grain boundaries and twin boundaries.

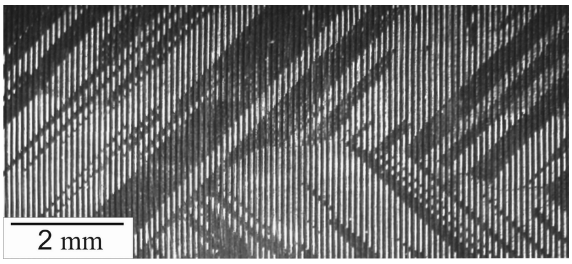

Fig. 7. Polycrystalline copper with identified twin boundaries after scratching the investigated polished surface by Vickers diamond pyramid indenter and additional oblique illumination. The scratches were applied with a normal load of 15 g with the distance between the scratches of 100 μm. Additional oblique illumination of the sample surface was applied at the angle of ≈ 10°.

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Новий підхід до виявлення меж зерен у результаті нанесення подряпин на поверхню полікристалічного зразка

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Експериментально отримано новий спосіб виявлення меж зерен у полікристалічних зразках. Підхід реалізується завдяки нанесенню на поліровану поверхню подряпин методом склерометрії й освітлення поверхні зразка пучком світла, який направлений під малим кутом до неї перпендикулярно напрямку нанесення подряпин. Запропонований підхід ґрунтується на анізотропії механічних властивостей кристалічного зразка. Встановлено, що профіль матеріалу зразка, витисненого на поверхню зерна при нанесенні подряпини, залежить від кристалографічної орієнтації зерна. Зображення в оптичному мікроскопі профілю матеріалу в області подряпини при косому освітленні поверхні зразка різко змінюється при перетинання меж зерен і ця зміна дозволяє точно визначити положення межі зерен.