Method and industrial equipment for the X-ray diffraction control of the monocrystalline materials in machine and instrument engineering

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Abstract. Design and characteristics of the domestic mobile X-ray diffraction unit used to control the orientation of monocrystalline materials are described. Its possibilities during the control of products used in aero engines and made from monocrystals: rods, seed plates, seed cones and individual areas on the surface of castings, where metallographically were identified subgrains, are shown.

1. Introduction

Achieving high efficiency of the modern aircraft engine, including increasing its operating temperature and service life, requires creation of the monocrystalline blades of a high pressure turbine, which eliminates the chance of destruction on the grain boundaries. This imposes specific requirements to the crystallographic orientation of the blades and to the disorientation of their substructure, which can be provided by the use of appropriate X-ray diffraction methods of orientation control, detection of subgrains and determining the magnitude of their disorientation [1, 2].

2. Methods and equipment

High-temperature nickel-based alloys used in the formation of monocrystalline structure of the castings of working blades of gas turbine engines [1] have a face-centered cubic structure. The technological process requires orientation and cutting of monocrystalline seeds, as well as control of the orientation of the main crystal and the disorientation of the subgrains in the finished blades. The error in measuring the orientation of monocrystals is allowed within 1° [2], although the deviation of the crystallographic direction from the geometric axis Z of the turbine blade is allowed within 10° [3]. The disorientation of the subgrains in the finished blade relative to the main crystal should not exceed 3–5° [4].

In practice the deviation of the subgrain orientation from the axis of the main crystal sometimes reaches tens of degrees [5]. For mass control of the serial monocrystalline products of this type, the measurement time should not exceed several minutes. Currently, the optimal characteristics and cost of the X-ray machine that meets these conditions have the installation PRDU “KROS” [6], developed by JSC “ELTECH-Med” (Saint Petersburg). The installation implements the symmetric Laue method in
reverse shooting with registration of the Lauegram using the screen with a photostimulable phosphor having 30×40 mm format. X-ray optics scheme of the PRDU “KROS” installation is shown in figure 1.

![X-ray optics of the PRDU “KROS” apparatus (L1, L2 – are laser pointers).](image)

**Figure 1.** X-ray optics of the PRDU “KROS” apparatus (L1, L2 – are laser pointers).

The sample may have any shape and any surface area. Positioning of the product and the radiation source is carried out by the means of three motorized linear-coordinate movers and two laser pointers. Exposure time is several tens of seconds. Digitization of the image with the subsequent transfer to the computer is made by a laser reader. Processing of the Lauegram and determination of the crystallographic orientation (CGO) of the monocrystal is performed using a specially developed program in an interactive mode [7]. The total time required to obtain a result is 3–4 min per sample.

Checking of the perpendicularity of the primary beam axis to the plane of the coordinate table and the correct measurement of the azimuthal orientation is performed using a control sample (CS) – cubic monocrystal in a holder with a flat rectangular base. Orientation of the monocrystal relative to the base of the holder is such that when placing CS on the coordinate table, the crystallographic axes <100> coincide with the axes of the device coordinate system XXYZ with high accuracy. Deviation of the beam axis from the normal to the plane of the table introduces a systematic error in the measurement results. Measurement of the CS can reduce this error to a value not exceeding two tenths of a degree.

The developed software allows to automatically locate the reflexes on the inverse Lauegram, determine their coordinates and convert inverse Lauegram into a stereogram. Program simulates the rotation of the polar complex of a cubic crystal around three coordinate axes. In the initial position, the axes of the polar complex <100> coincide with the axes XYZ, and the corresponding stereogram – with the standard grid 100. Rotations of the polar complex in real time are accompanied by updating of the model stereograms which are superimposed on the experimental stereogram in the working window of the program. Automatic comparison of the calculated spherical projection with the experimental one obtained from the Lauegram begins with an arbitrary choice of one of the strong reflexes by an operator.

The search for the parameters of the optimal combination of stereo patterns is performed using the simplex method. At each iterative step four parameters vary at three levels – three rotation angles around the XYZ axes and the distance from the sample to the detector plane. The process continues to find the minimum average matching error that characterizes the entire set of pairs of nodes. The finishing automatic combination of stereo patterns is accomplished with a reproducibility of about one tenth of a degree.
Practice shows that when working with samples of heat-resistant nickel alloys, the average error is 0.2–0.3°. When analyzing perfect crystals (diamond, germanium, silicon), the average error is close to 0.1°. This accuracy of measurement allows using one unit to control both seed rods and cast blades.

Both for the main crystal and for each of the subgrains, the program finds the orientation in a form of a matrix of guiding cosines of three mutually perpendicular crystallographic axes in the instrument coordinate system. When measuring the orientation of the main crystal, two resulting values and a table are displayed for the operator. Resulting values are as follows: crystallographic orientation of the axis [001] with respect to the instrument axis $Z$ and the azimuthal orientation of the axis [100] with respect to the instrument axis $X$. In the table for the main crystal, in addition to the two values of the CGO, the orientation of the axis [010] relative to the $Y$ axis is displayed (figure 2).

![Figure 2](image_url)

**Figure 2.** Working window of the “CGO-analysis” program.

To control the orientation of the identified using the metallographic method subgrains, the operator must shift the sample to place each subgrain under the incident X-ray beam and repeat the measurements. For the subgrains in a separate table the orientations are displayed both in the system of $XYZ$ axes of the apparatus and relative to the crystallographic axes <100> of the main crystal. The value of the maximum disorientation of the subgrain relative to the main crystal is also derived. In addition, the program presents a graphical result in a form of a picture, combining an experimental stereogram with a standard grid.

3. Conclusion

The results of operation of the PRDU “KROS” installation on the number of enterprises of aerospace industry of Russia showed that the inverse lauegram method with the registration on the screen with photostimulable phosphor and software processing of the diffraction patterns, realized in this unit, allows to provide hundred percent express control of the orientation of monocry stalline products made from high-temperature alloys under conditions of industrial production.

References

[1] Higginbotham G J S 1986 Materials Science and Technology 2 442–60
[2] Tikhomirova E A and Sidorin E F 2011 Bulletin of Samara state aerospace university 3 43–9
[3] Nikolaenko V A, Morozov V A and Kasianov N I 1976 Revue des Hautes International Température et des Réactantes 13 17–20
[4] Toloraya V N, Orekhov N G and Chubarova E N 2012 Foundry engineering 6 25–30
[5] Shishkareva L M and Kuzmina N A 2014 Proceedings of VIAM 1 6
[6] Potrakhov N N, Khayutin S G, Lifshits V A and Oses R 2015 Plant laboratory. Diagnostics of materials 8 27–30
[7] Oses R, Lifshits V A, Potrakhov E N and Potrakhov N N 2011 Certificate about the state registration of the computer program 2011614448