Communication

Determination of Crystal Orientation by Ω-Scan Method in Nickel-Based Single-Crystal Turbine Blades

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The article presents an assessment of the crystal perfection of single-crystal turbine blades based on the crystal orientation and lattice parameter distribution on their surface. Crystal orientation analysis was conducted by the X-ray diffraction method Ω-scan and the X-ray diffractometer provided by the EFG Company. The Ω-scan method was successfully used for evaluation of the crystal orientation and lattice parameters in semiconductors. A description of the Ω-scan method and an example of measurement of crystal orientation compared to the Laue and EBSD methods are presented.

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High-pressure turbine blades (HPT) in aircraft and land-based gas turbines operate under severe conditions. These include heavy mechanical loads, high temperature and oxidizing gas environments. Turbine blades are manufactured from nickel-based superalloys in an investment casting process, which usually involves application of the lost-wax casting method. A columnar grain as well as single-crystal structure increases their high-temperature creep resistance. This kind of structure is obtained as a result of directional solidification processes.[1–3] Single-crystal casting is a highly advanced process in terms of maintaining proper solidification conditions, preparation of wax models and ceramic shell molds. The wax model of the turbine blade consists of a starter block and grain selector. Their specific shape makes it possible to obtain a single-crystal structure of the cast. The spiral shape of the grain selector ensures growth of only one grain at its end.[4] It has been established that during the initial stage of the solidification process, nuclei grow in a columnar manner in liquid metal toward the spiral-shaped selector. During the second stage of solidification, differences in the withdrawal rate value and the influence of the selector geometry result in elimination of the majority of disoriented columnar grains on the walls. This process enables the growth of one grain and makes obtaining a structure with a homogeneous crystallographic orientation possible. In most cases, single-crystal turbine blades are obtained with [001] direction parallel to or slightly deviating from the blade load axis, usually designated as 'z.' High deviation leads to a decrease in creep resistance due to the high anisotropy of the mechanical properties. Worldwide aircraft engine manufacturers' quality criteria require the value of the deviation angle (α) to be lower than 15 deg.[5,6] The microstructure of single-crystal nickel-based superalloys consists of two main phase components: the γ phase, a solid solution of alloying elements in nickel, and γ', an Ni_3Al intermetallic phase. γ' has an ordered structure—$L1_2$, with a face-centered lattice—and is a main strengthening phase. The lattice parameters of the matrix (γ phase) and precipitates (γ' phase) have almost the same value: $a_0 = 0.356$ nm. High coherence of these phases allows classifying the structure of castings as single crystals, however with relatively low quality.[7–10]

Recently, in the gas turbine industry determination of the crystal orientation in single-crystal nickel-base superalloys has been conducted using Laue X-ray diffraction. This method provides information about the crystal orientation for the area of about 1.5 mm in diameter. The measurement of the α angle value in the casting takes about 30 minutes per measuring point. Despite the small measuring area, the obtained diffraction pattern can be difficult to interpret because of the high mosaicity of the superalloy casts. In the Research and Development Laboratory for Aerospace Materials, a novel diffractometer for the Ω-scan method was developed as an alternative to the Laue method. Prof. H. Bradaczek and the EFG Company have used the Ω-scan method for many years for the quick measurements of the crystal orientation of semiconductors.[11] This required the construction of a diffractometer with a rotation table and specially calculated diffraction slits for a measured material. Using a conventional X-ray source Ω-scan method delivers more precise automated results for the crystal orientation and lattice parameters and is a powerful, nondestructive tool for characterizing single-crystal superalloys casts.

In current research, the Ω-scan method is used to verify the crystal orientation and lattice parameter distribution of turbine blade surfaces. Additionally,

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the Laue method and electron back-scattered diffraction measurements were applied for comparison.

Single-crystal turbine blades made of a commercial CMSX-4 nickel-base superalloy were studied. The blades were manufactured at the Department of Material Science and Research and Development Laboratory for Aerospace Materials of Rzeszow University of Technology. The casting process was performed using the Bridgman method at a typical withdrawal rate of 3 mm/min.

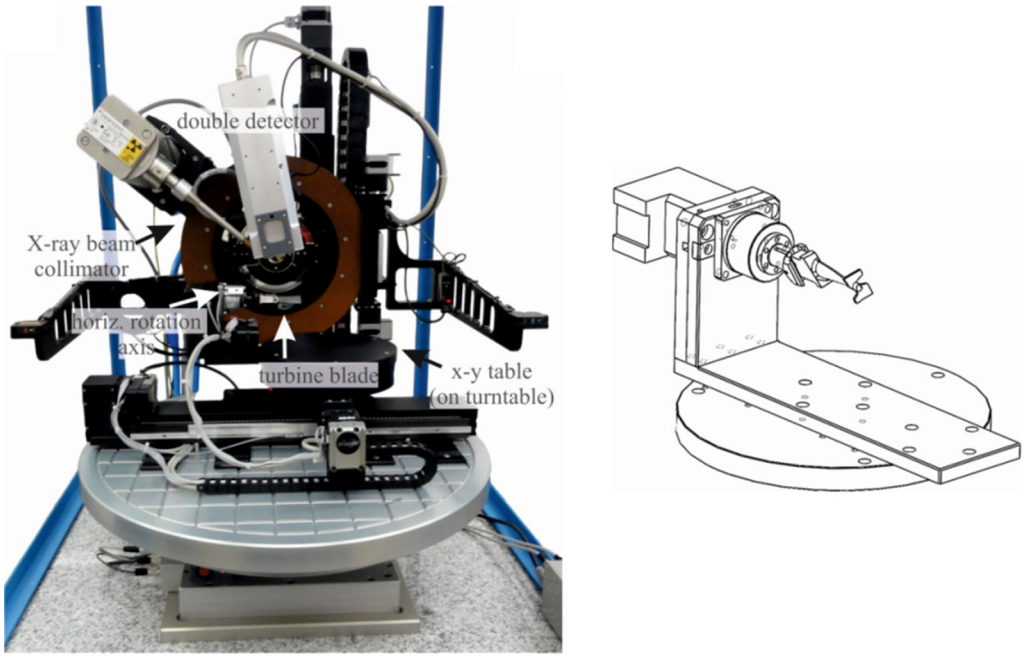
X-ray diffraction analysis was performed using a custom-built diffractometer (Figure 1(a)). The device was designed and manufactured by the EFG Berlin factory in cooperation with Rzeszow University of Technology and University of Silesia. The diffractometer is designed for automated determination of the crystal orientation on the whole turbine blade surface. The analyzed surface is scanned using an *x*–*y* table and a rotary axis (Figure 1(b)) by laser triangulation. Scanning (surface mapping) is necessary to create a map of the surface topography.

During the Ω-scan measurement, data collected in the surface mapping procedure are used to maintain correct alignment of the sample surface and detector. The crystal orientation is determined by means of the Ω-scan method so far applied for the single crystals used in electronics, *e.g.*, silicon, mainly with the generally known orientation.[12] The geometry of this method corresponds to the classical rotating-crystal method. The X-ray beam is reflected twice from the measured lattice planes inclined to the rotation axis. The angular positions of the reflections are measured and from these positions the azimuthal angle can be evaluated. From the lattice-plane angles, the orientation of the crystal lattice is determined. Diffraction peaks are recorded by one of two detectors during a full rotation of the sample depending on the rotation angle (Figure 2).

This method of measuring each point takes only a few seconds. The size of deviation angle (α) is calculated in reference to the center of the rotary axis. The X-ray source CuK_{α} and detector are in fixed positions. Slits in front of the detectors assure that only pre-selected reflections are measured. The area of the sample surface irradiated by the incident X-ray beam has a diameter of about 1 mm. The mapping can be performed in steps of 0.1–4 mm. Crystal orientation measurement on the curved element as a blade is possible using a rotary goniometer in the EFG diffractometer. The rotary goniometer provides constant blade height during measurement.

Determination of the crystalline orientation is based on diffraction peak identification. This is done by comparing the measured and calculated positions. Diffraction is performed in pre-selected points on the surface map. A higher number of selected points results in more accurate measurement of the crystalline orientation on the sample surface. Additionally, to save time, the crystalline orientation of unselected points can be interpolated.

The nickel-based superalloy crystalline orientation is defined by three angles similarly to the Euler's angles describing the relation between two Cartesian coordinate systems, that of the crystal lattice (*X*1, *X*2, *X*3) and that of the sample (**A′, B′, C′**) correlated to the measuring arrangement. The axis **A′** corresponds to the rotary axis; direction **B′** is perpendicular to it. The axes and orientation angles are shown in the stereographic projection (Figure 3(a)). The angle **α′** is the angle between the crystallographic main direction **X1**



(a) **(b)**

Fig. 1—Diffractometer for orientation measurement of single-crystal nickel-based superalloys. (*a*) Total view; (*b*) blade-mounting arrangement.

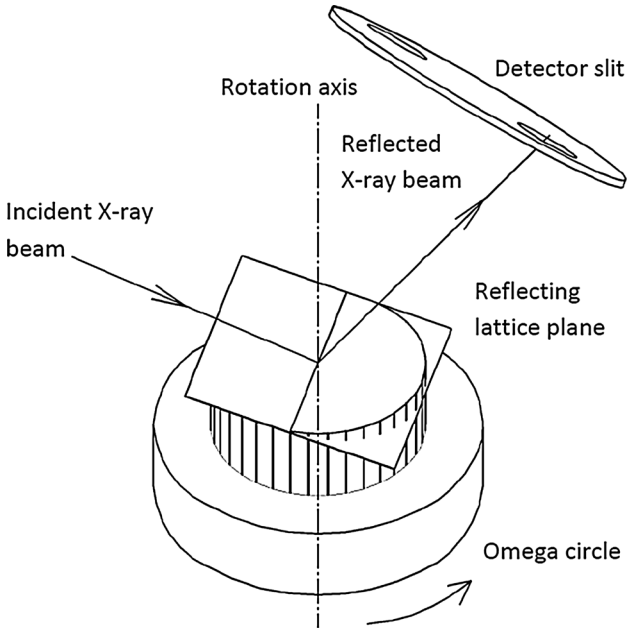


Fig. 2—Principle of the Ω-scan measuring method.

and the rotation axis **A′**. The angle ***β′*** describes the angle between the planes containing **A′** and **X1** and that containing **X1** and **X2** (*X*3 plane). The third angle γ' is measured between the second reference direction **C′** and the projection of the nearest crystallographic axis (**X3**).

These angles describe the orientation of a sample with the main axis parallel to the rotation axis, *e.g.*, measuring the cross section or any other plane cut through the blade.

The orientation of the turbine blade surface is determined as follows: The reference axis **A** of the blade is perpendicular to the rotation axis parallel to **B** (Figure 3(b)). The angles ***α***, ***β*** and ***γ*** are calculated from ***α′***, ***β′*** and ***γ′***, evaluated from the measurement data.

All angles (***α***, ***β***, ***γ***) can be measured at any sample point and presented in suited contrast pictures showing the distribution over the sample surface (Figure 4). The measurement error of the angle ***α*** and ***γ*** is on the order of 0.05 deg for a measuring time of 5 seconds; that of ***β*** (about 0.4) is larger depending on the value of ***α***. Laue method measurements were conducted from the flat part of root sections in back-reflection geometry using a copper anode. The Laue pattern was recorded on image plates. The electron backscatter diffraction (EBSD) investigations were conducted on a Jeol JSM-6480 scanning electron microscope (SEM) from the flat fragment of the root area. The SEM was operated at 25 kV of accelerating voltage. The specimens were placed at 20-mm working distance tilted to a 70 deg angle.

Mapping of the crystallographic orientation on the turbine blade surface by the Ω-scan method revealed the existence of two macroscopic grains going along the main axis of the blade. This can happened when a grain selection defect occurs in the selector area. The measured value of the ***α*** angle (deviation of [001] crystallographic direction from the withdrawal axis/load axis) on the surface of the turbine blade manufactured at a withdrawal rate of 3 mm/min was in the range of 3.1 to 9.3 deg (Figure 4(a)). However, we could distinguish that grain 1 had ***α*** values from 7.9 to 9.3 deg and grain 2 from 3.0 to 4.8 deg. Hence, this blade met the assumed criterion for the crystallographic orientation—the value of the deviation angle ***α*** was lower than 15 deg. Deviation of the ***β***-angle varied more because of the specifics of its calculations, which for small ***α*** values increase its sensitivity. Values of the ***β***-angle for the blade surface were in the range of 80-131 deg (Figure 4(b)). Grain 1 has ***β***-angle values between 80 and 85 deg and grain 2 between 116 and 131 deg. Angle γ—between the second reference direction C and the projection of the nearest crystallographic axis (*X*3)—was in range of −13.5 to 30.1 deg (Figure 4(c)) where grain 1 was in the range of 19.9 to 30.1 deg and grain 2 from −13.5 to −3.5. The total misorientation of the grains was about 37 deg. For the complete set of α, β and γ angle values (Table I), the minimum, maximum and delta differences were determined. Values of lattice parameters are the average of the γ and γ' phase lattice parameters and are similar to those in the literature data of from 3579 to 3587 Å (Figure 4(d)).[13]

The highest difference in value of crystallographic orientation was found near the trailing edge (grain 2) and at the end of the airfoil part of the turbine blade. This indicates that a complex subgrain structure and low angle boundaries are present in this area. The lattice parameter changes are mostly located on the thin trailing edge of the airfoil. The thin part of the airfoil has a different thermal gradient during crystallization process, which may induce the creation of defects. This can lead to a decrease in the mechanical properties and shorten the lifetime of the turbine blade. The small cross section of the airfoil's trailing edge induces the growth of secondary dendrite arms during the crystallization process, which are known to possess micro-subgrains. For direct comparison of the Ω-scan method results, Laue measurements and EBSD were additionally conducted on the blade surface. The typical Laue back-reflection method as well as EBSD method involves a flat surface for measurements. For this reason the crystallographic orientation and map of misorientation from both grains were measured on the flat root part of the blade (Figure 3(a)). Using the Laue method, the calculated values of α show a similar misorientation about 7.0 deg (grain 1) and 3.2 deg (grain 2) from the growth directions (Figure 5). The calculated misorientation of the grains was about 33.6 deg.

EBSD maps obtained from the grain boundary area show a similar total misorientation of the grains of about 34 deg (Figure 6). The boundary appeared to be located in the interdendritic region. The specific details of the grain selection failure show that the surviving grains have small misorientations in the growth direction (value α) and are mostly rotated to each other. The values of the α angle (φ_1 on EBSD maps) slightly deviated from that obtained by the Laue and Ω-scan methods because of the difficult mounting of the turbine blade on the sample stage.

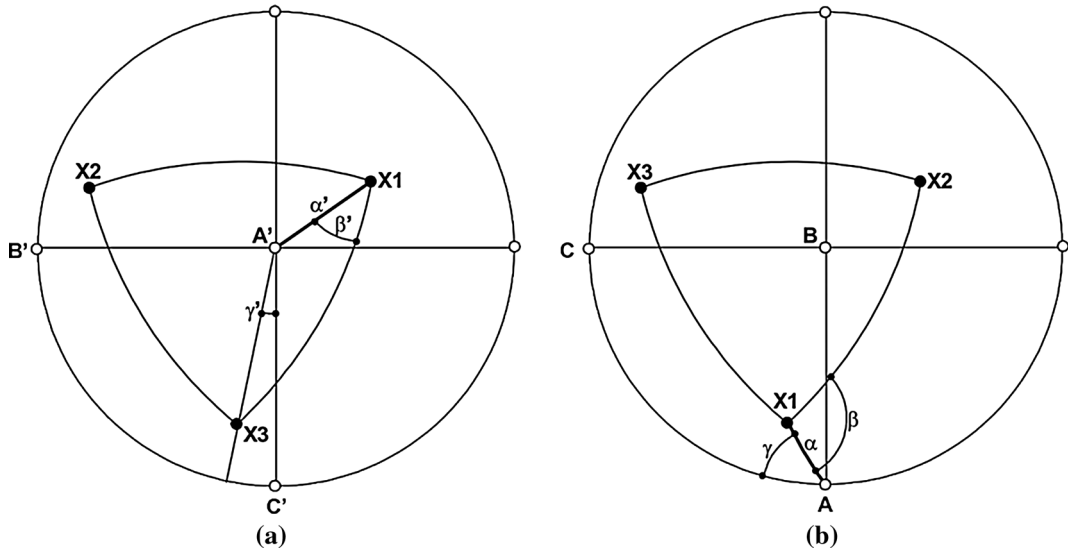


Fig. 3—Angles and directions describing the orientation of a cubic crystal (stereographic projections parallel to the turntable; for an explanation, see the text): (*a*) related to the turntable axis; (*b*) related to an axis in the turntable plane.

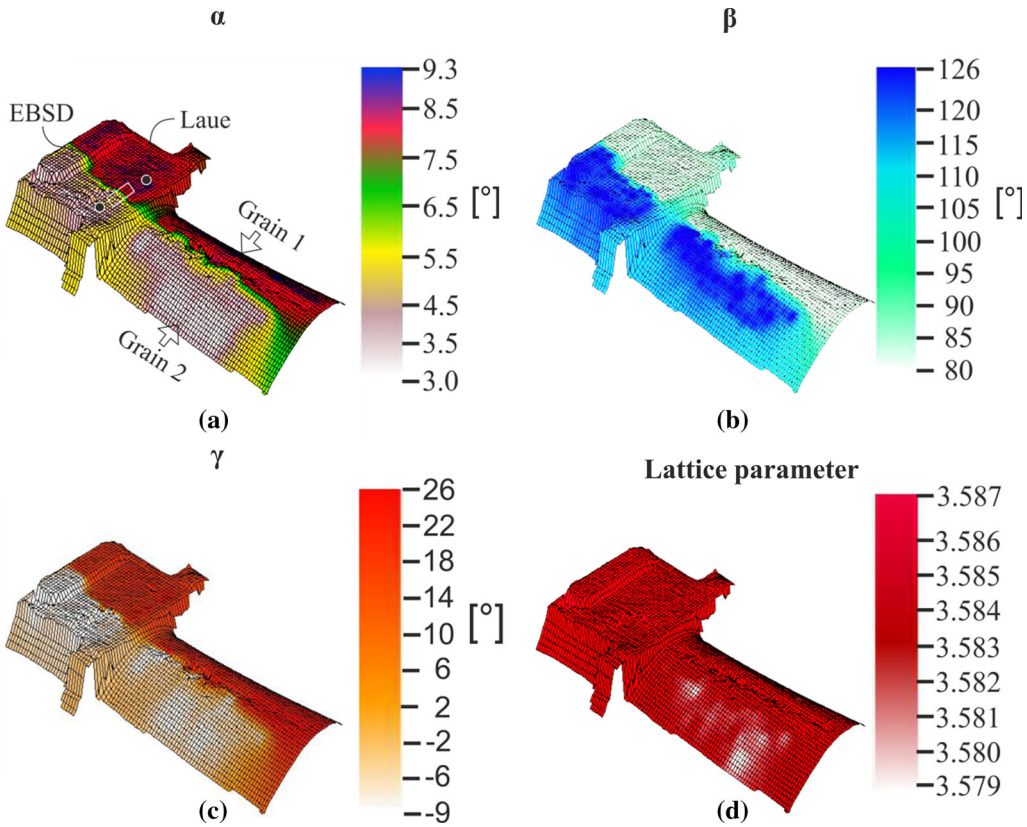


Fig. 4—The distribution of the value of angle α (*a*), β (*b*) and γ (*c*) and lattice parameter (*d*) on the surface of the blade airfoil and root of the single-crystal blade manufactured at the withdrawal rate of 3 mm/min.

An X-ray diffractometer (OD-EFG Berlin) was used to evaluate the crystallographic orientation of a single-crystal turbine blade manufactured with a CMSX-4 nickel-based superalloy. Crystallographic orientation was described by three deviation angles (α, β, γ) from crystallographic directions [100], [010] and [001]. These crystallographic directions corresponded to reference axes *x*, *y* and *z*, related to the crystallization directions.

This novel method and special diffractometer are particularly dedicated for the assessment of the perfection degree of single-crystal blades made of nickel superalloys. They allow determination of the basic

Table I. The α, β and γ Angles Values from the Ω-Scan Measurements

	Whole Blade			Grain 1			Grain 2		
Angle	Min (deg)	Max (deg)	Δ (deg)	Min (deg)	Max (deg)	Δ (deg)	Min (deg)	Max (deg)	Δ (deg)
α	3.05	9.26	6.21	7.85	9.26	1.41	3.05	4.77	1.72
β	79.67	130.92	51.25	79.67	84.53	4.86	116.85	130.92	14.07
γ	−13.53	30.08	43.61	19.92	30.08	10.16	−13.53	−3.48	10.05

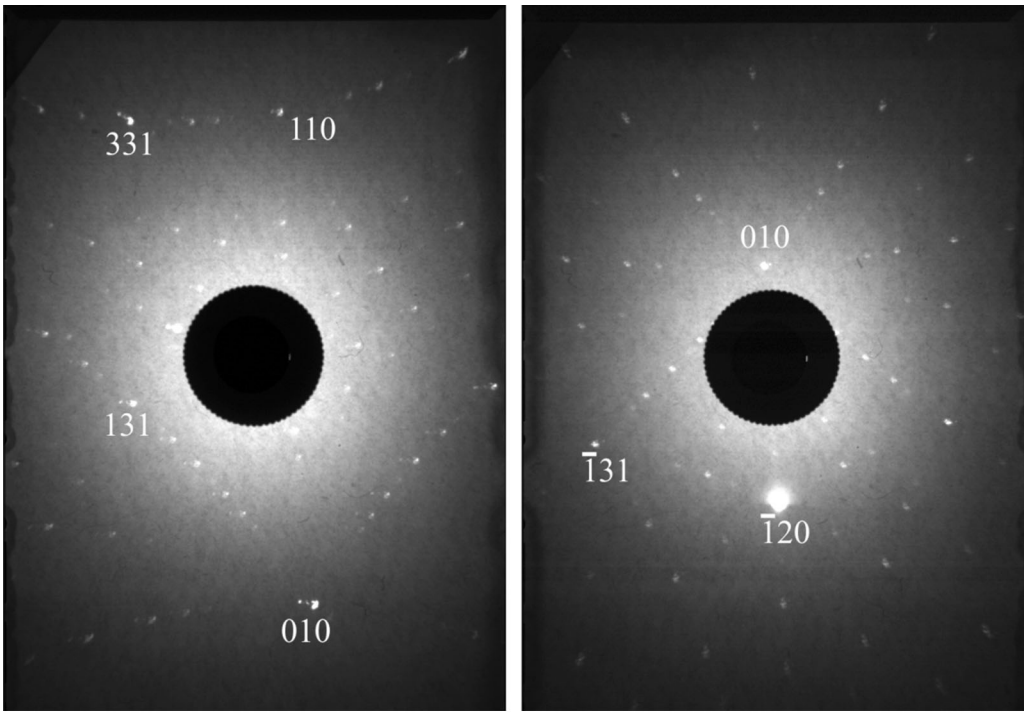


Fig. 5—Images of Laue diffraction for (*a*) grain 1 and (*b*) grain 2.

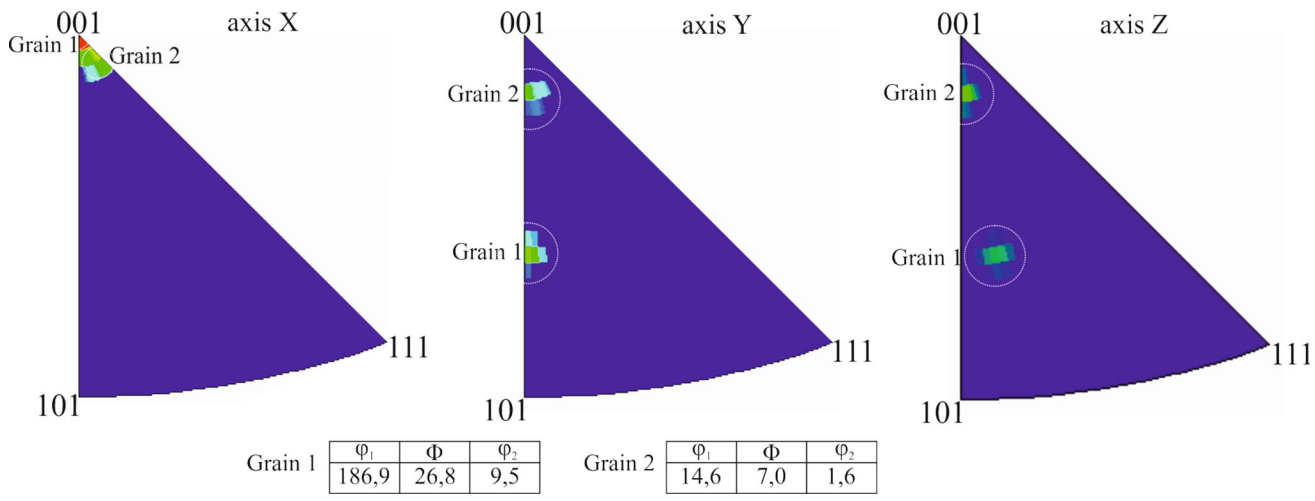


Fig. 6—Inverse pole figures of both grains measured on the root surface.

parameter of the single-crystal turbine blade—the angle between the withdrawal direction of the blade and [001] direction.

The industrial criterion for assessment of the single-crystal perfection degree applied in most standards is the value of the deviation angle α, usually requiring the value of α to be lower than 15 deg.

Currently applied criteria for assessment of structure perfection for single-crystal castings—only by the deflection angle of particular areas of blade casting related to their reference axes and main crystallographic directions—will be moreover analyzed by the angles β and γ.

The analysis of results obtained using a novel diffractometer (OD-EFG) allows stating that the manufactured blade castings meet the industrial standards and criteria for evaluating structure perfection applied by worldwide aircraft engine manufacturers. The OD-EFG diffractometer is capable of measuring the crystal orientation nearly fully automatically with high precision, making it suitable for industrial application. The results of the Ω-scan method were verified by other orientation imaging techniques, such as Laue X-ray diffraction and EBSD. It has been experimentally confirmed that the results do not differ between methods. The Ω-scan and OD-EFG diffractometer allow determining the crystal orientation from the whole blade surface and are faster than the other methods.

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REFERENCES

1. R.C. Reed: *The Superalloys Fundaments and Application*, Cambridge University Press, Cambridge, 2006, pp. 122–30.
2. A. Onyszko: *Solid State Phenom.*, 2013, vols. 203–204, pp. 169–72.
3. D. Szeliga, K. Kubiak, J. Suchy, G. Jarczyk, and J. Sieniawski: *Mater. Eng.*, 2013, vol. 1 (34), pp. 7–13.
4. A. Royer, P. Bastie, and M. Veron: *Acta Mater.*, 1998, vol. 15 (46), pp. 5357–68.
5. K. Kubiak, A. Onyszko, J. Sieniawski, W. Bogdanowicz, and A. Nowotnik: *Mater. Eng.*, 2010, vol. 3 (31), pp. 622–24.
6. S. Seo, J. Lee, Y. Yoo, C. Jo, H. Miyahara, and K. Ogi: *Metall. Mater. Trans.*, 2011, vol. 10 (42), pp. 3150–59.
7. H. Pang, H. Dong, R. Beanland, H. Stone, C. Rae, P. Midgley, G. Brewster, and N. D'Souza: *Metall. Mater. Trans.*, 2009, vol. 7 (40), pp. 1660–69.
8. O. Ola, O. Ojo, P. Wanjara, and M. Chaturvedi: *Metall. Mater. Trans.*, 2012, vol. 3 (43), pp. 921–33.
9. F. Wang, Y.D. Mao, S. Bogner, and A.B. Polaczek: *Metall. Mater. Trans.*, 2016, vol. 1 (47), pp. 76–84.
10. W. Bogdanowicz, R. Albrecht, J. Sieniawski, K. Kubiak, and A. Onyszko: *Solid State Phenom.*, 2012, vol. 186, pp. 135–138.
11. H. Berger, H.A. Bradaczek, and H. Bradaczek: *J. Mater. Sci. Mater. Electron.*, 2007, vol. 1 (19), pp. 351–55.
12. H. Berger: *J. Phys. Arch.*, 2004, vol. 4 (118), pp. 37–42.
13. B. Dubiel: *Microstructural Changes During Creep Resistance of Single-Crystalline Nickel-Base Superalloys*, AGH University of Science and Technology Press, Kracow, 2011, pp. 75–84.