Two methods of studying structure perfection of single crystal nickel-based superalloys

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Abstract. The article presents the comparison of two methods: classical X-ray topography and the mapping method based on the modern automatic X-ray OD-EFG diffractometer. Both methods were applied to study the crystal orientation of turbine blades of single crystal industrial CMSX-4 superalloys. The solidification of a hollow assembly structure for 5 various blades was carried out by the Bridgman method at the Research and Development Laboratory for Aerospace Materials at Rzeszow University of Technology using an ALD Vacuum Technologies vacuum furnace.

Both methods involve X-ray diffraction but they present misorientation of the turbine blades in two different ways. The mapping method allows for faster analysis of misorientation of any surface of the blades but the research resolution of this method is lower.

Introduction

The aviation development level forces the application of heat-resisting and high-temperature creep resistant materials of complex chemical composition and sophisticated microstructure for blades and components of the turbofans hot part. Increasing the operation temperature of aircraft engines is possible as a result of application of turbine rotor blades made of nickel or cobalt superalloys of equiaxed structure (EQ), directionally solidified (DS) or single crystal (SC). Nickel and cobalt based superalloys are used for components of the hot part of aircraft engines, operating in complex load conditions: combustor, gas turbine and compressor blades. These are units deciding about the power and efficiency of an aircraft turbine engine and of an industrial stationary gas turbine.

Nickel CMSX-4 superalloy based on single-crystal microstructure (SC) is the material, which features the best properties during the operation at high temperatures. It contains about 70% of cubic phase γ′ (Ni₃Al) microcrystals oriented in the same way, between which there is the lattice of phase γ and the range of other phases. In macroscopic experiments (Laue or classic X-ray topography) X-ray diffraction of such alloys has the same character as in single-crystals.

The third generation of SC alloys is used in turbine engines and now the development work is ongoing on the fourth generation of these alloys, containing alloying additions from the group of platinum metals. These materials will be used in new-generation turbine engines and in industrial gas turbines. The change of the chemical composition and of the way of aircraft engines components manufacturing caused the tenfold creep stress increase within 60 years [1-4].

The assessment of crystalline quality, including the crystallographic orientation, is a very important element of manufacture of single crystal blades casts made of nickel single crystal superalloys. Superalloys’ properties change depending on the crystallographic direction taken for studies. Hence the assessment of single crystal casts via determination of their crystallographic orientation is the basis for determining high temperature mechanical properties. It has been assumed
that the value of the angle of direction \([001]_y\) deviation from the blade axis cannot exceed 15°. It has been determined that an increase in this angle results in decreased monocrystals’ creep resistance [5-7]. This deviation can be examined by mapping and by X-ray topography method. The aim of the study was to compare the usefulness of both methods in the analysis of misorientation of single-crystal blades.

**Experimental**

The OD-EFG diffractometer is equipped with a copper tube and two detectors – dynamic scintillation counters (Fig. 1). A 3D image of the studied object is obtained by laser beam scanning. Then a map (distribution) of crystallographic orientation on the studied surface of the single crystal is created. The determination of crystallographic orientation on a 3D surface is especially significant due to technological reasons – it creates a non-destructive method for the crystallographic orientation studies of single crystal blades of aircraft engine turbines (Fig. 2). 2D cross-sections of single crystal blades were examined in the experimental part. The presentation of Fig. 2 in the paper (3D image) demonstrates possibilities of the newly produced OD-EFG diffractometer.

**Fig. 1.** OD-EFG diffractometer for full crystallographic orientation of single crystal blades made of nickel superalloys: a) general view, b) goniometer for single crystal blades

**Fig. 2.** Crystallographic orientation for a single crystal blade made of CMSX-4 nickel superalloy – withdrawal rate of 3 mm/min – distribution of angle \(\alpha\) values on the surface. T - the axis of the blade, which is the direction of crystallization.
The crystallographic orientation (value of angle $\alpha$ – the angle between direction [001] and the direction of blade) was examined at fixed points on the blade surface, creating from them a map of crystallographic orientation distribution of the blade surface. The beam was 1 mm in diameter and such resolution was assumed for ‘mapping’. Fragments of Laue diffraction patterns – selected reflections – were obtained at each measuring point from polished side surfaces of the blades. On their basis, using computer software, the crystallographic orientation (angle $\alpha$) was determined. Basic parameters of measurements were: the detector distance from the specimen – 80 mm, the exposure time – 5 s for one point, copper tube, radiation $K_{\alpha}$, $\lambda = 1.542\text{Å}$.

X-ray topograms were obtained using the modified Auleytnr method with specimen’s oscillation relative to the vertical axis [8]. Co$K_{\alpha}$ radiation originating from an X-ray lamp with a microfocus 30 µm in diameter was used. Topograms were recorded on an AGFA Structurix D7 FW film.

**Results and discussion**

The results of hitherto research on polyphase single crystal casts provided the basis to design an automated diffractometer to study the distribution of crystallographic orientation on the whole surface of single crystals. The OD-EFG diffractometer was manufactured by the company EFG Berlin. Parameters of the designed and manufactured instrument take into account the needs of the Research and Development Laboratory for Aerospace Materials at Rzeszow University of Technology. The OD-EFG diffractometer has characteristics of known and used methods: Laue and Auleytnr (X-ray topography). It is equipped with a goniometer enabling performance of studies on 3D surfaces. This makes the diffractometer unique not only within the country. Additional features of the OD-EFG diffractometer as compared with traditional Laue diffractometer comprise: the time of measurement – approx. 100 times shorter; the detector is coupled with the computer and the software processing the obtained results during the measurement; the information about the crystallographic orientation applies to the whole area of studied single crystals – in the Laue method – only the area 1.5 mm in diameter (beam diameter); crystallographic orientation is determined for 3D objects, e.g. blades surface – when using the Laue method this is very difficult.

Moreover, the diffractometric method enables a higher accuracy of the measurement by the application of laser calibration of the instrument and of computer-controlled goniometer. Characteristic X-rays are used in this method, while in a traditional Laue diffractometer – usually a continuous radiation.

Fig. 3. Single crystal blade made of CMSX-4 nickel superalloy – a) X-ray topogram (reflection 002 of $\gamma'$ phase), Z – oscillation axis, b) map of crystallographic orientation for angle $\alpha$ prepared using the OD-EFG diffractometer
The performed analysis of the results obtained by means of OD-EFG diffractometer and X-ray topography (Fig. 3) allows stating that: the OD-EFG diffractometer resolution is worse than in the Auleytnert method, but the interpretation of the results is easier (Fig. 3). For example, the OD-EFG diffractometer resolution does not allow for observing M bands originating from dendrites visible on X-ray topogram (Fig. 3a). Furthermore the time of measurement for the OD-EFG diffractometer is approx. 50 times shorter as compared with the Auleytnert method; X-ray topography is obtained only from a flat (2D) surface, while the OD-EFG diffractometer enables obtaining a 3D image; maps of crystallographic orientation of individual areas produced by means of the OD-EFG diffractometer give the value of crystallographic misorientation against the selected direction, while an X-ray topogram specifies mutual rotation of neighbouring areas of the single crystal.

Summary

The comparison of research results obtained with the use of the X-ray OD-EFG diffractometer and X-ray topography allows stating that: the OD-EFG diffractometer resolution is lower than X-ray topography, however the interpretation of the results is less complicated. The measurement time for the OD-EFG diffractometer is ca. 50 times shorter than in case of X-ray topography. X-ray topography is obtained merely from a flat (two-dimensional) surface yet the OD-EFG diffractometer makes it possible to gain three-dimensional image. The crystal orientation maps of particular areas created with the OD-EFG diffractometer depict crystal misorientation values with regard to a selected direction; on the other hand, X-ray topograms define mutual rotation of neighboring areas of the single crystal.

To conclude, both research methods have their benefits, however for industrial purposes it’s better to use the OD-EFG diffractometer. The reason is short measurement time. Additionally, OD-EFG diffractometer measurements are not destructive. The research resolution – crystal orientation maps – obtained with the OD-EFG diffractometer is lower, though it’s sufficient for industrial applications.

References