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Measurements of γ/γ' Lattice Misfit and γ' Volume Fraction for a Ru-containing Nickel-based Single Crystal Superalloy

X.P. Tan[1)], *J.L. Liu*[1)], *X.P. Song*[2)], *T. Jin*[1)†], *X.F. Sun*[1)] *and Z.Q. Hu*[1)]
1) Superalloy Division, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China
2) Analysis and Testing Division, Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China
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A conventional X-ray diffractometer has been used to determine the γ/γ' lattice misfit and γ' volume fraction for a Ru-containing nickel-based single crystal superalloy at room temperature. The rocking curve was used to characterize the distribution of subgrains. The diffraction peaks obtained by ω–2θ scan were used to determine the γ/γ' lattice misfit and γ' volume fraction. A three peaks fitting model was proposed. The peak fitting results are in good agreement with the model. The X-ray diffraction results indicate that the nickel-based single crystal superalloy was not a perfect monocrystalline material, which is comprised of many subgrains; and each subgrain also consists of large numbers of mosaic structures. In addition, two anomalous reflection phenomena were found during the experiment and discussed with respect to their occurrence and impact on the measurement. The experimental results show that the γ/γ' lattice misfit and γ' volume fraction will be various at the different regions of its dendritic microstructure. The average γ/γ' lattice misfit and γ' volume fraction of the experimental alloy are approximately –0.2% and 70%, respectively. Furthermore, the γ' volume fraction calculated by atom microprobe (AP) data is also basically consistent with the experimental results.

KEY WORDS: **Nickel-based superalloy; Microstructure; X-ray diffraction; Lattice misfit; γ' volume fraction**

1. Introduction

Nickel-based single crystal superalloys can be treated as a special kind of bi-phase metal-matrix composite by *in situ* reaction synthesis. It consists of coherent γ matrix and γ' strengthening phase with $L1_2$ ordered structure. Ideally, the γ' phase with cubic morphology is homogeneously distributed in the γ matrix after full heat treatment. In addition, nickel-based single crystal superalloys are also typical coherent secondary phase strengthening single crystal composite. The relationship between γ and γ' at the interface is characterized by a lattice misfit that can be larger or smaller[1,2]. Therefore, the γ/γ' lattice misfit and γ' volume fraction are the two most significant structural parameters for this material.

As known, it is difficult to precisely measure the γ/γ' lattice misfit usually defined as $\delta = 2(a_{\gamma'} - a_\gamma)/(a_{\gamma'} + a_\gamma)$ for a given nickel-based single crystal superalloy, where a_γ and $a_{\gamma'}$ are respectively the lattice constants of the γ and γ' phases. The corresponding reasons are the low values of δ, the segregation of refractory element, the mosaic structure in crystals and the distorted lattice cells caused by coherent stress at the γ/γ' interfaces[3]. It can be easily presumed that the γ/γ' lattice misfit varies in different locations of a single crystal superalloy. Therefore, methods to obtain a local γ/γ' lattice misfit are critical. To obtain an average γ/γ' lattice misfit, the X-ray diffraction (XRD) technique was most frequently applied previously[4–7]. For the measurements of γ/γ' lattice misfit by XRD, the first question is to locate the exact diffraction planes and the other is to accurately separate the strongly overlapping γ and γ' peaks. Obviously the peak fitting is the key factor for

† Corresponding author. Prof., Ph.D.; Tel.: +86 24 23971757; E-mail address: tjin@imr.ac.cn (T. Jin).

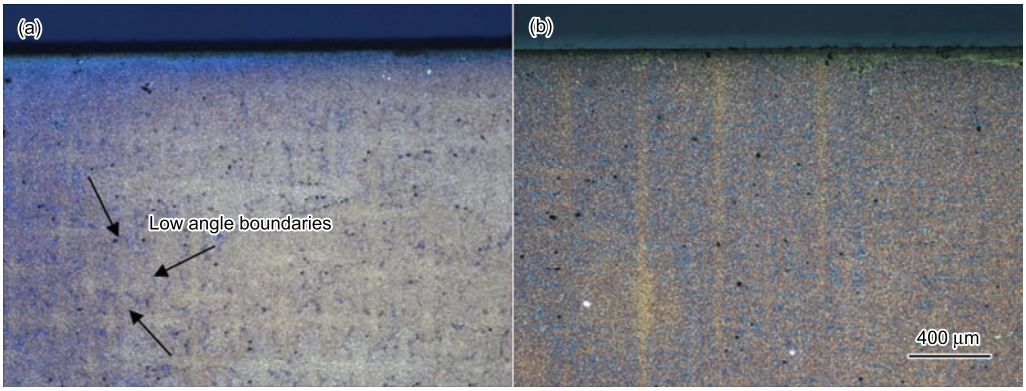

Fig. 1 Optical micrographs of (a) TS and (b) LS specimens

the experimental results. In order to promote resolution by reducing the instrumental broadening as much as possible, either a modified high-resolution diffractometer or synchrotron radiation combined with a special triple crystal diffractometer were widely used. Moreover, a multi-purpose sample stage was also necessary to locate the plane with maximum diffraction intensity[8–12].

In single crystal superalloys, the volume fraction of γ' ($f_{\gamma'}$) is very large usually, reaching 70% or more. By assuming that the area fraction equals to the volume fraction, image analysis from scanning electron micrographs was the most frequently used method to determine the γ' volume fraction. Great care was required when using this method, since the results are sensitive to the γ' particle morphology, due to the high interface/volume ratio. Afterwards, measurements were also made by neutron diffraction or synchrotron radiation[13,14], based on the diffraction kinematical theory, *i.e.* the integrated intensity of a reflection is directly proportional to the material which diffracts. The advantage of this method is that large numbers of precipitates are included in the measurements, regardless of their size and shape, and *in situ* measurements can be made during elevated temperature. Another important method is the time of flight atom microprobe (AP), which enables $f_{\gamma'}$ to be calculated directly from the compositions of γ, γ' and overall alloy with higher accuracy[15]. The major disadvantage is the extreme localization of the zone analyzed, particularly when the alloy was not perfectly homogeneous. Last but not least, theoretical method by thermodynamic software was also involved in some work[16], which might show great discrepancy with experimental measurements at most cases.

The purpose of this work is to develop a simple and reliable method for the determination of the γ/γ' lattice misfit and γ' volume fraction in a Ru-containing single crystal superalloy by using an X-ray diffractometer and a detailed characterization of its microstructure. To our knowledge, there exists no work which correlates the measurement of the γ/γ' lattice misfit with the γ' volume fraction. Therefore, this work will give an insight into the measurements of the γ/γ' lattice misfit and γ' volume fraction with better accuracy.

2. Experimental

2.1 Specimens preparation

The chemical composition (in wt%) of the alloy investigated was as follows: 6Al, 12Co, 20(Cr, W, Mo, Ta), 5Re, 3Ru and balance Ni. Single crystal bars (16 mm in diameter and 22 mm in length) were fully heat treated by 1325°C/8 h + 1335°C/16 h, air cooling, 1150°C/4 h, air cooling and 870°C/24 h, air cooling. Owing to its high refractory elements additions (up to 23 wt%), the alloy was hardly homogenized in short time. As a result, the cross-like dendrites both in the transverse sections (TS) and longitudinal sections (LS) can be clearly observed with the naked eyes after etching (as seen in Fig. 1). According to the dendrites growth direction, specimens for (001) TS and (100) or (010) LS were cut by spark erosion. The specimens had dimensions of 10 mm×5 mm×1 mm. The spark-cut surfaces were ground and then mechanically polished.

2.2 X-ray diffraction

The XRD profiles were recorded by a conventional Rigaku D/MAX 2500 X-ray diffractometer with Cu$K\alpha$ radiation at 50 kV/300 mA. This instrument was set up for Bragg-Brentano geometry with a line focus and a graphite monochromator between specimen and detector. The radiated area is about 10 mm×5 mm. Due to the larger reflection region, the experimental results represent statistical averages. The intensity profiles of {004} reflection were collected to measure γ/γ' lattice misfit and γ' volume fraction. Besides, the intensity profiles of {003} superlattice reflection were also collected to determine the position of γ' sub-peaks in {004} reflection profiles. The step scans were made at 0.02 deg. per step and the counting time was adjusted to make sure that