Microtextural characterization of as-cast and hot press-deformed TiN whiskers in TiNw-AlNp/Al composite *

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Abstract The local distribution of preferred orientation and misorientation in TiN whiskers in TiNw-AlNp/Al composite system is investigated using backscattering electron diffraction combined with a scanning electron microscope. These preliminary results demonstrate the utility of this new automated technique for characterizing local textures in composite materials using backscattering electron diffraction. The composite investigated comprises TiN whiskers in a polycrystalline aluminum matrix. The local textures of samples before and after deformation were measured. The misorientation distributions show a breakdown of the monocrystal of TiN whisker in the deformed composite material relative to the starting material and an increase in small angle misorientations.

Keywords: Al matrix composite, gas-liquid reaction, microtextural characterization, TiN whisker.

Researchers have been paying much attention to the composites produced by *in situ* reaction owing to their many advantages, such as simple fabrication process, low cost of fabrication, and excellent and controllable mechanical properties. Recently, the studies on the *in situ* metal matrix composites (MMCs) are focused on making full use of the dispersion strengthening of *in situ* intermetallics and ceramic particles, and making enough volume fraction reinforcements with thin size distributed in thinned matrix. Although preferred orientation in some pure metals such as Cu, Al and Ti during deformation has been well investigated and also reproduced in the models of polycrystalline plasticity, the study on misorientation texture, which is an important part of the analysis of interface microstructure in alloys and metal matrix composites (MMCs), has received far less attention. Advances in the measurement of microtextures using backscattered electron Kikuchi diffraction patterns (BEKPs) in the scanning electron microscope[1] allow us to study the texture of misorientations between grains. The introduction of automated analysis of BEKPs[2,3] enables us to perform the quantitative studies on misorientation distribution. In this work, TiN whisker and AlN particles reinforced Al matrix composites were made by the gas-liquid reaction and the rapid solidification technique. And then an orientation image microscope of TexSEM Laboratories Inc. (TSL) and its related techniques were applied to the investigation of microtextural characterization of as-cast and hot-press-deformed TiN whisker in TiNw-AlNp/Al composite.

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1 Experimental procedure

1.1 Fabrication

Al-4.6Ti-3Mg was melted in an induction furnace in a temperature range between 1 000 and 1 200℃, and subsequently nitrogen gas was introduced into the molten aluminum alloy in a form of turning gas-mixing, so titanium and aluminum atoms reacted with active nitrogen atoms [N] to form TiN and AlN crystal nuclei. Owing to the turning gas and electromagnetic mixing, the reacting alloy melt exhibited a homogeneous distribution of fine *in situ* formed nuclei which moved with the mixing alloy liquid. Under the above condition, the *in situ* nuclei gradually grew to become microcrystal particles of sizes ranging from 20 to 100 nm. In the following reaction process the particles did not grow obviously. While enough volume fraction reinforcements were produced and the liquid alloy had not become obviously sticky, the treatment of degassing was carried out, and then the reacted alloy melt was allowed to solidify in a flat steel model cooled with cycling water at its bottom. Finally the TiNw-AlNp/Al composite was produced. The as-cast billets were extruded at 470℃ with 80% reduction in area. Samples were machined to test various mechanical properties such as tensile and to study the microstructure of materials.

1.2 Metallography and orientation measurements

Metallography studies of as-cast and hot-press-deformed TiN whiskers in TiNw-AlNp/Al composite were carried out using a Philips XL30/TMP scanning electron microscope. The microtextural characterization was carried out using a TSL orientation image microscope (OIM) and the measurement sequence was as follows. Orientations were measured using automatic technique based on computer analysis. A sample was mounted in the scanning electron microscope (SEM), and the sample normal was directed 70°with respect to the incident electron beam. A collimated electron beam was focused on the surface of the sample. The interaction of the electron beam with the crystal lattice produced backscatter-diffracted electrons that fluoresced onto a phosphor screen mounted in the microscope chamber. A high-grain video camera transmitted the pattern produced on the phosphor screen to a computer where the pattern was automatically indexed. The computer also controls the movement of a stage in the microscope. Thus, the sample can be translated in the SEM under computer control so that the lattice orientations can be automatically measured at spatially specific locations on the sample. Generally, the computer is programmed so that the translations form a regular array of measurement points on the sample. The spatial resolution of the technique is less than 1 μm, the angular resolution is less than 0.5°, and the cycle time is approximately 1 s per orientation measurement. Using this technique, approximately 11 000 orientation measurements were made on two kinds of samples obtained before and after hot-press-deformation.

2 Results and discussion

Structural compositions of both as-cast and hot-press-deformed *in situ* composite fabricated by the liquid-gas reaction and rapid solidification are α-Al, AlN and TiN, which are evaluated by X-ray diffraction as illustrated in Ref. [1]. Fig. 1 shows an image of the as-cast TiNw-AlNp/Al composite recorded by SEM.

Figure 2 shows a pole figure of whisker in as-cast TiNw-AlNp/Al composite formed by mapping the image qualities of the corresponding electron back-scatter diffraction pattern (EBSP) at every point in the measurements. To investigate the texture of material near the TiN whiskers and Al matrix, the measurement data were partitioned. Firstly the data from TiNw were removed from the data set by excluding data with both low image quality and low confidence index values. The pole figures in Fig. 2 show a strong and simple texture. However, the peaks in these pole figures simply correspond to the monocrystals.

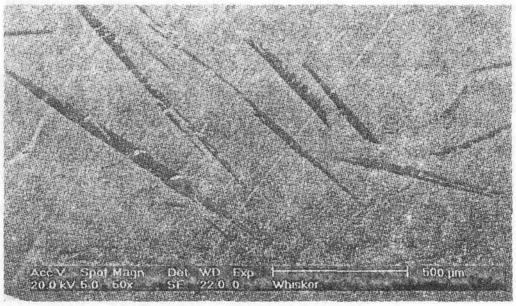


Fig. 1 Microstructure of *in situ* TiN-AlN/Al composites in the as-cast state.

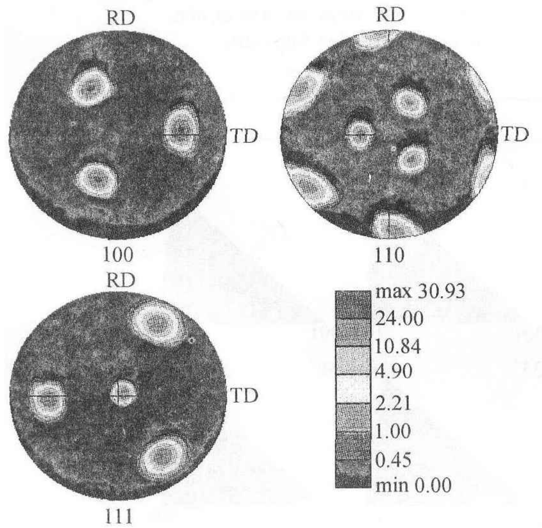


Fig. 2 Pole figures for TiN whisker in TiN-AlN/Al composite in state of as-cast.

OIM data can also be used to characterize the distribution of misorientation in a polycrystalline sample[4~7]. For each pair of neighboring points in the OIM scanning the misorientation is calculated. If a misorientation exceeds a given value (in this case, 5°), then it is used in the calculation of misorientation distribution. The orientation distribution function (ODF) and misorientation distribution function (MDF) are shown in Figs. 3(a) and 3(b) respectively.

The orientation texture is presented in this figure using an axis/angle representation[8]. For any two orientations there is a common axis. The misorientation can then be defined by the common axis and the angle of rotation which will bring the two orientations into coincidence with one another. In the plot in Fig. 3, the distribution of the rotation axes is represented in each section by the projection of the axis onto the plane in a manner similar to that used for pole figures. According to the test results, TiN whisker is certainly monocrystal.

SEM image of TiN whiskers in *in situ* TiN-AlN/Al composites in the hot-pressed state is shown in Fig. 4, in which the horizontal direction corresponds to the compression direction of the sample.

The pole figures for the hot-pressed sample are shown in Fig. 5. In these pole figures, the direction associated with the center of pole figures corresponds to the direction of extension in the hot channel-die compression test and is normal to the plane on which the orientations were measured. The vertical direction corresponds to the compression direction of the sample and the horizontal direction is the constrained or transverse direction of the sample. The pole figures have been rotated slightly, 10°

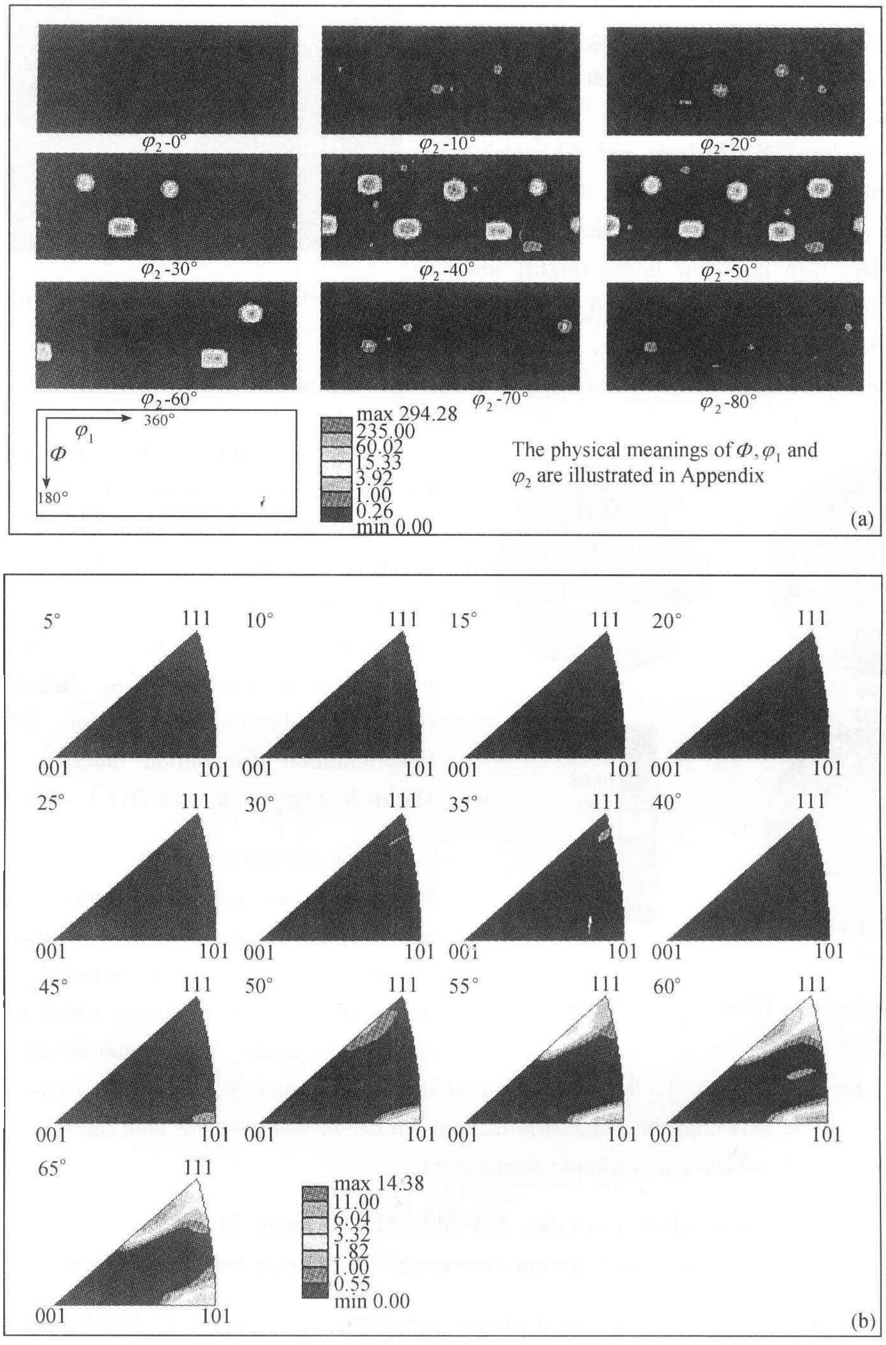


Fig. 3 Orientation distribution function (ODF) (a) and misorientation distribution function (MDF) (b) of TiN whisker in TiN-AlN/Al composite in as-cast state.

about the transverse axis and 3° about the compression axis. These rotations bring peaks in the (111) and (100) pole figures into coincidence with the centers of the two symmetrical texture components in the pole figures. As for the as-cast sample, the measurements for the TiN whiskers in Al matrix were excluded from the pole figure calculation.

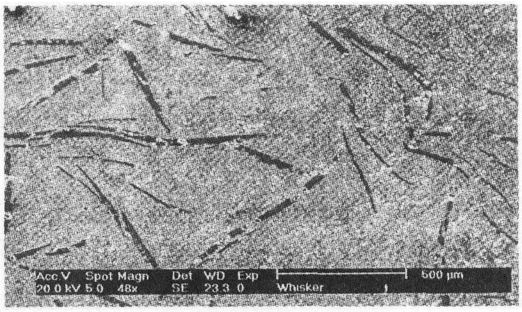


Fig.4 Microstructure of the hot-pressed composite.

The ideal measurement results within 0° ~ 80° in ODF and 5° ~ 65° in MDF are shown in Fig. 6 (a) and (b). The {111} type components are shown in thicker color and {100} types are shown in lighter color.

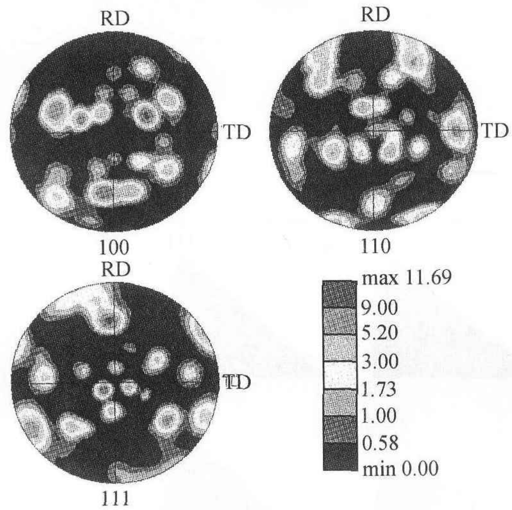


Fig. 5 Pole figures for TiN whisker in TiN-AlN/Al composite in hot-pressed state.

The misorientation texture is plotted in axis/angle space in Fig. 6(b). With an increase in angle, the percentage of boundaries in the microstructure is decreased[9]. However, the peaks associated with the broken TiN whiskers represent the prominent features in the misorientation distribution. The plot in Fig. 6(a) also indicates that small angle misorientations have increased in number as the intensity in the 10° plot section is much stronger than that in the same plot section for the TiN whisker in TiN-AlN/Al composite in the hot-pressed state. This could be considered as relating to the introduction of dislocations into the matrix material by deformation.

3 Conclusions

(i) *In situ* TiN whiskers and AlN particles reinforced Al composite can be made by the reaction between N_2 and Al-Ti alloy liquid and rapid solidification.

(ii) Electron diffraction results indicated that the local textures in the TiN whisker of TiN-AlN/Al composite in the as-cast state are very simple, i.e. TiN whisker is monocrystal.

(iii) The distribution of local textures in the TiN whisker of TiN-AlN/Al composite in the hot-pressed state composite is nearly random.

(iv) The plot also indicates that the number of small angle misorientations has increased as the intensity in the 10° plot section is much stronger than that in the same plot section in hot-pressed state. This might be related to the introduction of dislocations into the matrix material by deformation.

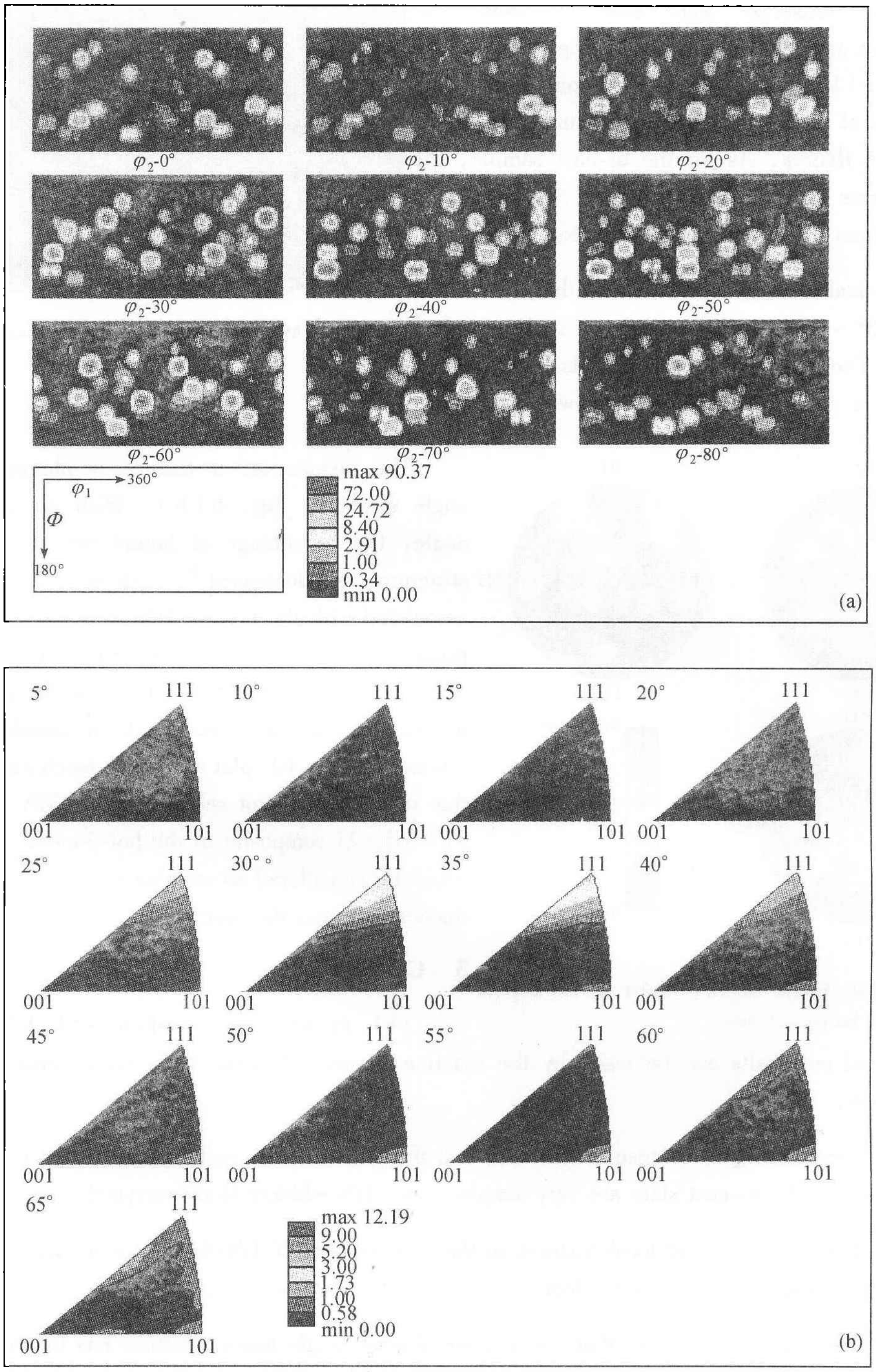
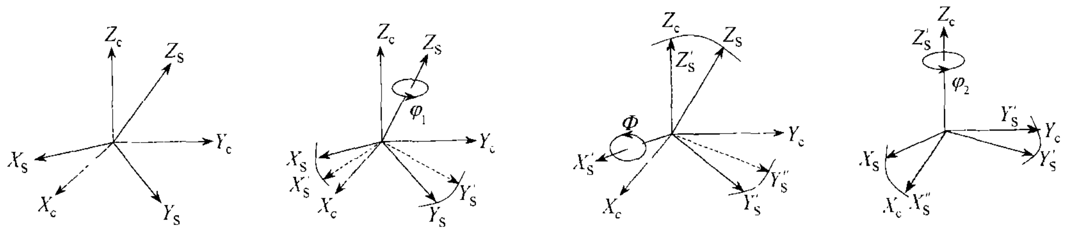


Fig. 6 Orientation distribution functions (ODF) (a) and misorietation of distribution functions (MDF) (b) of TiN whisker in TiN-AlN/Al composite in hot-pressed state.

Appendix

The physical meaning of Euler angles Φ, φ_1 and φ_2 in Figs. 3(a) and 6(a) is illustrated in the flowing picture. And X_c, Y_c and Z_c are axes of the TiN crystal; and X_s, Y_s and Z_s are axes of the specimen.



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