

## MORPHOLOGY AND MICROSTRUCTURE EVOLUTION OF $\Delta$ PHASE DURING CROSS WEDGE ROLLING OF GH4169 ALLOY

<sup>1,2</sup>GAN Hongyan, <sup>1</sup>CHENG Ming, <sup>1</sup>SONG Hongwu,  
<sup>1</sup>CHEN Yan, <sup>1</sup>ZHANG Shihong, <sup>3</sup>Vladimir Petrenko

<sup>1</sup>Institute of Metal Research, Chinese Academy of Sciences;

<sup>2</sup>School of Materials Science and Engineering, University of Science and Technology of  
China; <sup>3</sup>Physical-Technical Institute of the National Academy of Sciences of Belarus

*Optical microscope (OM) and electron backscatter diffraction (EBSD) were applied to observe the constant section shrinkage phase morphology and microstructure of the cross wedge rolling GH4169 superalloy under solid solution state. The phase morphology evolution and the microstructure impact during cross wedge rolling was studied. The results show that the  $\delta$  phase gradually bends, twists and cuts under the rolling force so that it changes from acicula to short rod or granule. With the increase of the section shrinkage, the spheroidization degree increases and the dynamic recrystallization of GH4169 alloy during cross wedge rolling is promoted by the existence of the  $\delta$  phase. The pinning effect of the relative grain boundaries inhibits the growth of the dynamic recrystallized grains and the average grain size of aged specimen is  $2.84\mu\text{m}$  with 50% cross section shrinkage, while the average grain size of the solid solution specimen is  $25.3\mu\text{m}$ .*

**Keywords:** GH4169 Alloy, Cross Wedge Rolling,  $\delta$  Phase, Microstructure, Dynamic recrystallization

### 1 Introduction

Cross wedge rolling is a material-efficient and net-shape metal forming process, which is widely used in the production of shaft parts [1-3]. At present, some achievements have been made in the theoretical research and technological application of cross wedge rolling forming. Zhao[2] et al. discussed the mass loose position of the aluminum alloy core in multi-wedge cross wedge rolling and the force variation rule along X, Y and Z directions at the center of the rolled parts was obtained with the width and forming angle. Peng [3] et al. used the multi-wedge cross wedge rolling process to produce the thick wall hollow shaft of large and long thickness then obtained the effect of the rolling parameters with 42CrMo4. Dong [4] et al. studied the interface slip phenomenon in plate cross wedge rolling process by numerical simulation method and the effects of the friction coefficient, forming angle and section shrinkage were analyzed.

However, there is no reports focusing on the microstructure evolution, the effect of  $\delta$  phase on the microstructure evolution and the  $\delta$  phase morphology evolution law of GH4169 alloy under cross wedge rolling. Zhang [5,6] et al investigated the metal flow regu-

larity, strain and temperature distribution, rolling and die parameters of GH4169 alloy during cross wedge rolling process by finite element method and the microstructure evolution was also studied. Zhu [7] et al studied the change rule of the rolling force and other technological parameters under cross wedge rolling of GH4169 alloy. Chen [8] et al analyzed the effect of rolling parameters on the interfacial slip of GH4169 alloy plate during cross wedge rolling. Lin [9] et al investigated the effect of forming temperature, strain rate and strain on microstructure evolution and dynamic recrystallization of Ni-based alloy during hot forming by EBSD technique. Zhang[10] et al learned the evolution of  $\delta$  phase in GH4169 alloy during Delta process and came to the conclusion that  $\delta$  phase changed from lamella to granule in the isothermal compression process of low strain rate under the combination of deformation fracture and dissolution fracture. Naya [11] et al studied the microstructure evolution of GH4169 alloy under large strain. Yuan[12] et al analyzed the effect of  $\delta$ -relative GH4169 alloy on hot forming. In this paper, the microstructure evolution of solid state and aged state GH4169 alloy under cross wedge rolling is studied, as well as the effect of  $\delta$  phase evolution and section shrinkage of aged GH4169 alloy workpiece, providing theoretical basis for producing GH4169 alloy aero-engine blade blanks by cross wedge rolling.

## 2 Experimental methods

The material is commercially available high quality GH4169 alloy cylinder forging rod of sample size  $\Phi 40 \times 100$  mm. The chemical compositions (mass fraction, %) are shown in Table 1. In order to eliminate the effects of residual stress, strain, segregation and second equivalence in the original specimens, the samples were first treated by solution treatment (1040°C, 40 min, water cooling) and  $\delta$  phase aging (915°C, 24 h, water cooling). The cross wedge rolling was carried out on the H630-roller cross wedge rolling machine (roll diameter 630 mm) and the die parameters are as followed: stretch angle  $\beta = 5^\circ$  and forming angle  $\sim 28^\circ$ . The rolling parameters were designed as 940°C, roll speed  $\sim 12$  r/min, and cooled to room temperature immediately to preserve the deformed structure.

**Table 1**

**Chemical compositions of GH4169 alloy (wt.%)**

C	Ni	Cr	Nb	Mo	Ti	Al	B	Si	Mn	Co	Fe
0.027	53.74	17.58	5.35	3.01	0.98	0.52	0.0025	0.009	0.07	0.40	Bal

The cross wedge rolling specimens were cut along the axial line, then mechanical grinding and polishing, followed by chemical etching, using HCl + H<sub>2</sub>O<sub>2</sub> (volume fraction) as the corrosion solution and observed under the Axiovert200MAT inverted metallographic microscope and SSX-550 scanning electron microscope (SEM) of ZEISS respectively. The specimen was electrolytic polished by 80% CH<sub>3</sub>CH<sub>2</sub>OH + 20% HClO<sub>4</sub> (volume fraction), then observed under FEI Nano SEM Nova 430 field emission scanning electron microscope to conduct EBSD technique, with the scanning voltage 20 kV, scanning step 0.5  $\mu$ m, using the Oxford Instrument's HKL Channel 5 EBSD System to extract the data information.

## 3 Experimental results

### 3.1 Microstructure of GH4169 alloy after different heat treatment processes

The microstructure of GH4169 alloy after solution treatment is homogeneous equiaxed crystal with a small amount of twins (Fig. 1), and the average grain size is about 80  $\mu$ m. Fig. 2 shows the microstructure of GH4169 alloy treated by  $\delta$ -phase aging.  $\delta$  phase precipitated at the grain boundary, the twin boundaries and  $\gamma$  phase matrix. Morphology of the precipitated  $\delta$  phase is lamella, and the average grain size is about 100  $\mu$ m.

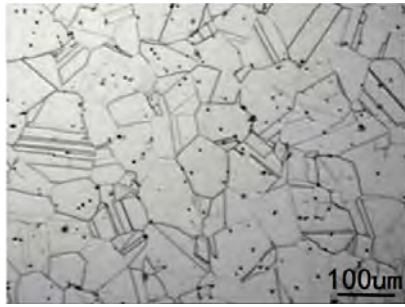


Fig. 1 Microstructure of solid solution

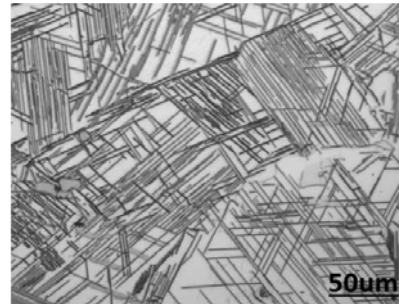


Fig. 2 Microstructure of aged GH4169 alloy  
GH4169 alloy

### 3.2 Phase Morphology of GH4169 Alloy during Cross Wedge Rolling with Different Section Shrinkage

Fig. 3 shows the microstructure and different section shrinkage of aged GH4169 alloy with  $\delta$ -phase under rolling temperature of 940°C. From Fig. 3a, 30% cross-sectional shrinkage indicates that the original lamellar  $\delta$  phase has been bent, twisted, and fractured to some extent and a small amount has been transformed into short rod or granule, while the  $\delta$  phase is still lamellar. When the surface shrinkage is 38% (Fig. 3b), the original lamellar  $\delta$  phase has mostly broken into short rod-like or granular phase while only a few lamellar  $\delta$  phase is found locally in the microstructure, which presents a axial distribution. When the shrinkage ratio of the cross section is 43% (Fig. 3c),  $\delta$  phase continuously breaks leaving no lamellar  $\delta$  phase of different length, short rod and grainy shape with different bending degree. When the shrinkage ratio of the cross-section increases to 50%, the  $\delta$  phase has completely changed to a granular phase and mainly disperses in the matrix in the form of spherical dispersion (Fig. 3d).

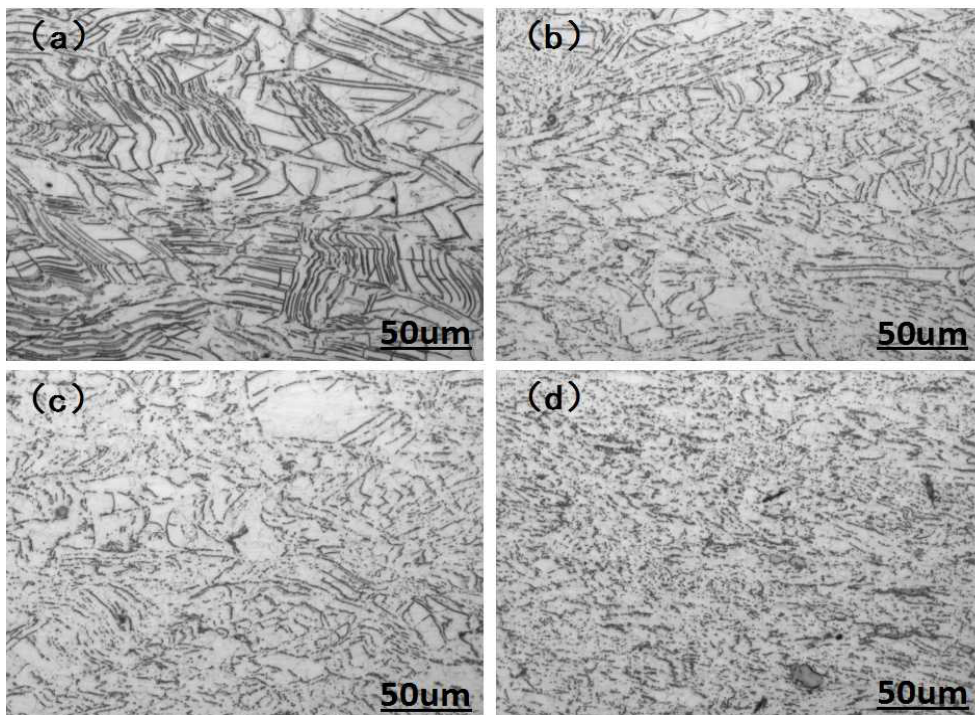


Fig. 3 The  $\delta$  phase morphology of GH4169 alloy wedge rolling pieces with different section shrinkage:  
a – 30%; b – 38%; c – 43%; d – 50%

### 3.3 Microstructure of GH4169 alloy under cross wedge rolling

#### 3.3.1 Microstructure of Cross Wedge Rolling GH4169 Alloy Parts under Different Heat Treatment Conditions

Fig. 4 shows the surface microstructures of the cross wedge rolling parts observed by the metallographic microscope of  $\delta$  phase aged GH4169 alloy with the section shrinkage of 50% and the rolling temperature of 940 °C. It can be seen that dynamic recrystallization takes place in the alloy with different heat treatment states during cross wedge rolling. The average grain size (2.84 $\mu$ m) of the  $\delta$  phase aged specimen in Fig. 4b is significantly smaller than the average grain size (25.3 $\mu$ m) of the solid solution wedge rolling specimen in Fig. 4a. This is because the  $\delta$  phase shatters into granular particles and  $\delta$ -relative grain boundaries pinning at grain boundaries inhibiting the grain growth.

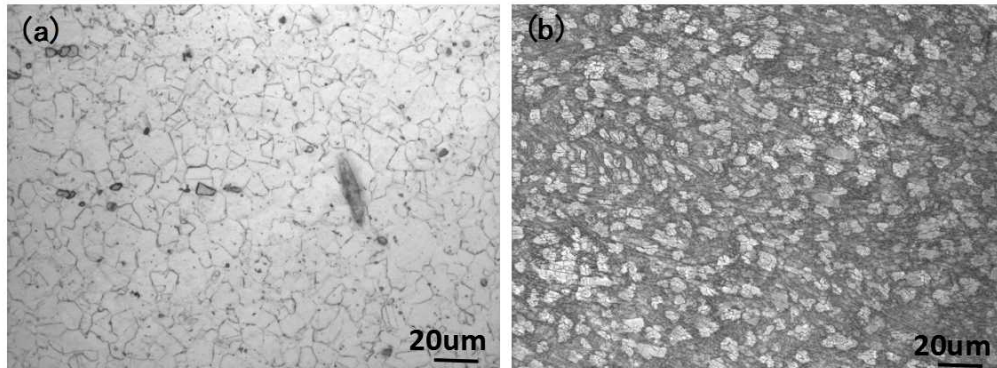


Fig. 4 Microstructure of GH4169 alloy with 50% section shrinkage in solution and phase aging:  
*a* – Solid solution; *b* –  $\delta$  phase ageing states

Fig. 5 shows the orientation image of GH4169 alloy under cross wedge rolling with 30% cross section shrinkage reconstructed by Channel 5 software. The coarse black and white fine lines are large angle grain boundaries ( $>15^\circ$ ) and small angle grain boundaries ( $<15^\circ$ ) respectively. It can be seen that there are many small angular grain boundaries in the deformed large grains in solid solution GH4169 alloy after cross wedge rolling. At  $\delta$  phase aged state, the small angular grain boundaries are distributed at the grain boundary and the amount is very small.

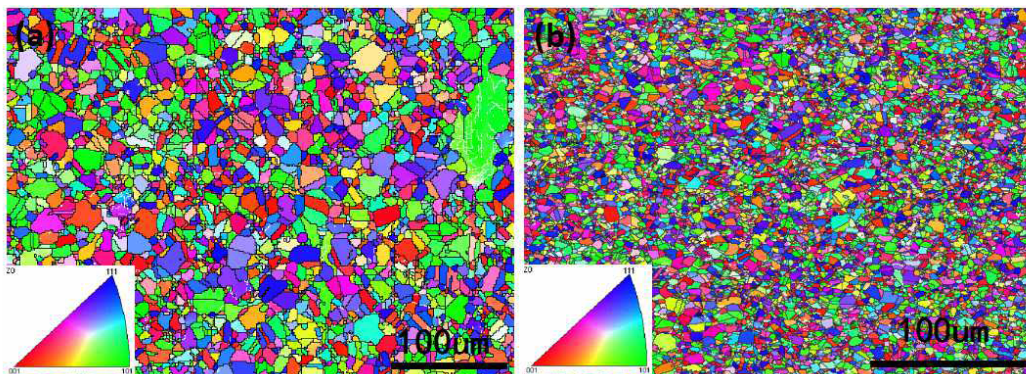


Fig. 5 Orientation diagram (OIM) of cross wedge rolling parts of GH4169 alloy under different heat treatment states:  
*a* – Solid solution; *b* –  $\delta$  phase ageing states

#### 3.3.2 Microstructure of aged GH4169 alloy at different section shrinkage

Fig. 6 shows the microstructure of  $\delta$ -phase aged GH4169 alloy after cross wedge rolling at different section shrinkage. The microstructure of cross wedge rolling is dynamic re-

crystallization grain and deformed initial large grain when the cross-sectional shrinkage is 30% and 38%. The initial large grain recrystallization volume fraction increases with the increase of section shrinkage and the complete dynamic recrystallization of GH4169 alloy during cross wedge rolling was achieved when the shrinkage ratio reaches 50%.

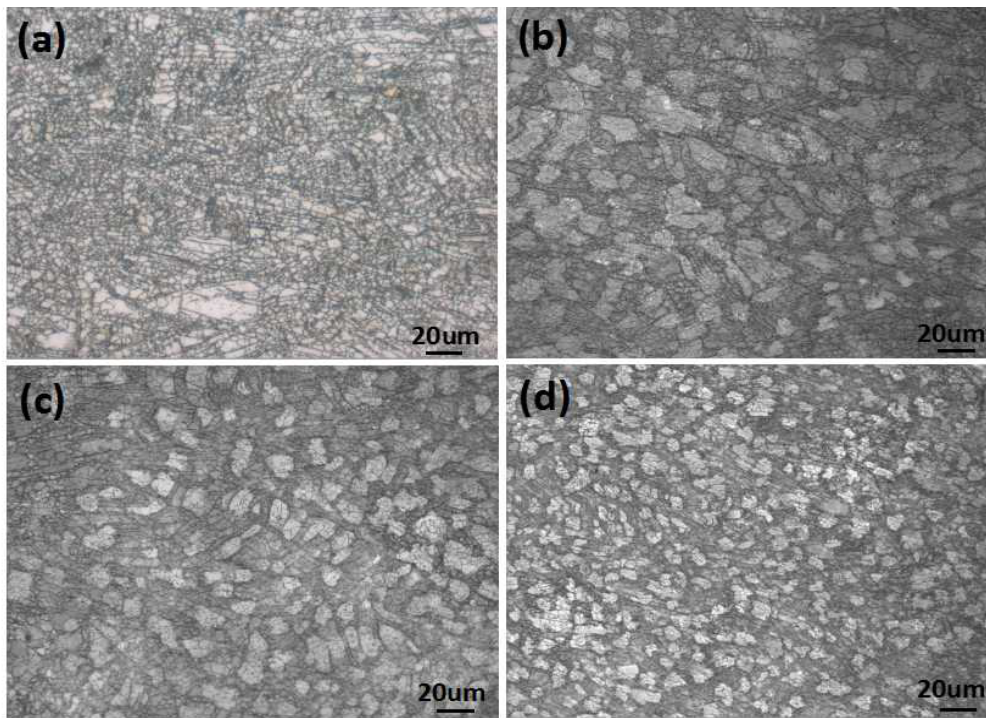


Fig. 6 Microstructure of  $\delta$  phase aged GH4169 alloy cross wedge rolling parts under different shrinkage:  
*a* – 30%; *b* – 38%; *c* – 43%; *d* – 50%

## 4 Analysis and discussion

### 4.1 Evolution of $\delta$ phase morphology during cross wedge rolling

The morphology evolution of  $\delta$  phase of GH4169 alloy by cross wedge rolling is similar to the  $\delta$  phase of evolution observed by Zhang [13] et al. in the isothermal compression of GH4169 alloy. The evolution of  $\delta$  phase is from lamella to short rod and granule during high temperature deformation. However, the  $\delta$  phase spheroidization has its own characteristics in the process of cross wedge rolling. This is related to the different state of the rolling pieces in the process of isothermal compression and cross wedge rolling (Fig. 7).

It can be seen from Fig. 7 that the specimen is only subjected to uniaxial compressive stress during isothermal compression while the specimen is under alternating load during cross wedge rolling. In the wedge section and the stretch section, the surface of the rolled piece in contact with die is subjected to three-dimensional compressive stress, while the center of the rolled piece in contact with the die and the surface of the rolled piece are subjected to two-dimensional tensile stress and constant compressive stress. On the other hand, the surface and the core of the rolled piece are only subjected to the axial tensile stress along the surface of the rolled section, since  $\delta$  phase and matrix  $\gamma$  phase are noncommutative relation [14], and  $\delta$  phase is a hard brittle phase relative to the matrix  $\gamma$  phase. In the continuous rotation process, the matrix  $\gamma$  phase takes precedence and the  $\delta$  phase is not easily deformed with the matrix  $\gamma$  phase. Stress concentration is easy to occur near the lamellar  $\delta$  phase then bending and torsion will occur under the effect of tri-directional alter-

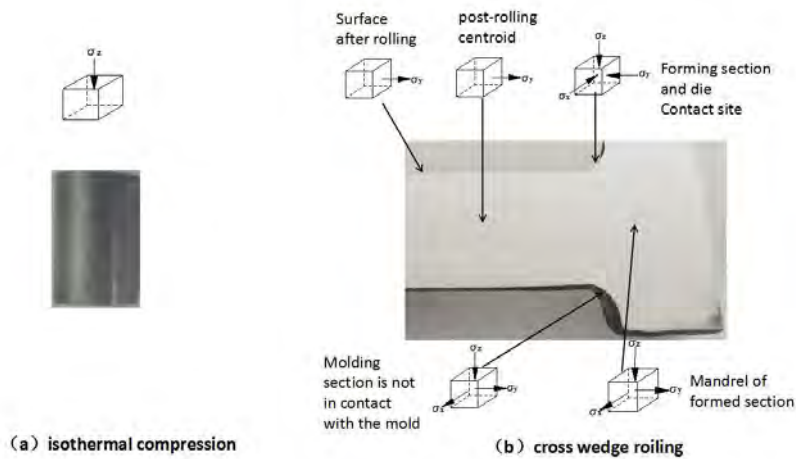


Fig. 7 Stress state analysis of rolled piece in the process of isothermal compression and cross wedge rolling

nating load including compressive stress, tangential force and axial force. When the fracture limit of the lamellar  $\delta$  phase reaches, it will break. In addition, during the deformation process, the sub-grain boundary or the high dislocation density region in the lamellar  $\delta$  phase will increase due to the effect of deformation, which can promote the dissolution fracture of lamellar  $\delta$  phase.

#### 4.2 Effect of Microstructure Evolution of $\delta$ Phase under Cross Wedge Rolling

In the deformation progress, the motional dislocations produced in the matrix in the deformed area are plugged around the lamellar  $\delta$  phase, resulting in the increase of dislocation density  $\rho$  around the  $\delta$  phase. The formation of high dislocation density region is due to the plug accumulation around the phase. At the same time, the existence of lamellar phase can reduce the nucleation incubation period of dynamic recrystallization and increase the nucleation site of dynamic recrystallization, so that to promote the occurrence of dynamic recrystallization. Under the same deformation condition, the dislocation density in the region of high dislocation density of pre-precipitate  $\delta$  phase can reach the critical dislocation density of dynamic recrystallization and reduce the critical strain of dynamic recrystallization of the alloy under the small deformation. The dislocation density and deformation distortion energy in the nucleation or grain of recrystallization are smaller than in the part without dynamic recrystallization. The recrystallization nucleation or grain boundary will migrate to the region of the high dislocation density in the non-recrystallization part. The lamellar  $\delta$  phase is spheroidized in the rolling process, and the spheroidized  $\delta$  phase is mainly distributed at the grain boundary (Fig. 8). The spheroidized  $\delta$ -relative grain boundary pinning inhibits the grain growth and refines the grain size. Under the same deformation condition, the dynamic recrystallization grain size decreases with the increase of  $\delta$  phase content in the alloy.

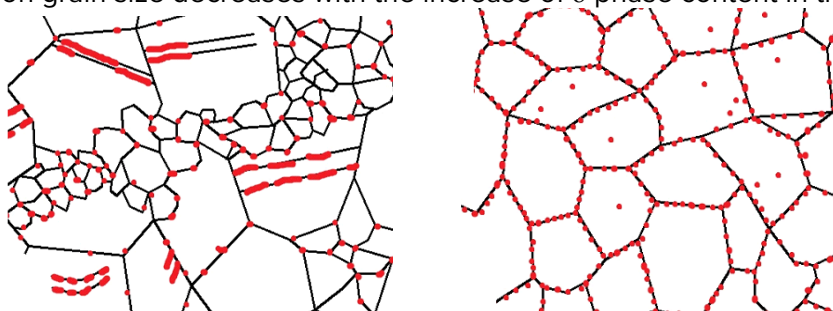


Fig. 8 Spheroidization and distribution of  $\delta$  phase

## 5 Conclusion

Through bending, torsion and fracture mode in the cross wedge rolling process of aged GH4169 alloy,  $\delta$  phase morphology changed from the initial lamella to the short rod or granule, and with the increase of the section shrinkage, the degree of  $\delta$  phase spheroidization increased, and became completely spheroidized when the section shrinkage reached 50%.

The presence of  $\delta$  phase can promote dynamic recrystallization of GH4169 alloy during cross wedge rolling. The pinning effect of grain boundary inhibits the growth of recrystallized grain.

The dynamic recrystallization of  $\delta$  phase of aged GH4169 alloy under cross wedge rolling is more sufficient with the increase of the section shrinkage.

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