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ARTICLE

Microstructure Evolution of Inconel 617 Alloy During Subzero Treatment

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Abstract: The relationship of microstructure evolution with subzero treatment time and number of Inconel 617 alloy was studied. The results indicate that subzero treatment has an obvious influence on the microstructure of Inconel 617 alloy. With the increase in subzero treatment time, the grain size decreases. With the increase in subzero treatment number, the grain size increases gradually, and the high stress is retained at the boundaries of the refined grains. The lattice constant varies inversely with the grain size. The simple and complex carbides of MC, M_6C and $M_{23}C_6$ are precipitated from the samples after subzero treatment, which leads to the accumulation of dislocations. The geometrically necessary dislocation density of the samples increases after subzero treatment for 24 h, and decreases significantly with two times of subzero treatment for 24 h. In addition, after subzero treatment, the rotated cube texture and rotated copper texture transform to brass texture, P texture and goss texture.

Key words: Inconel 617 alloy; subzero treatment; grain refinement; geometrically necessary dislocation; microtexture

Inconel 617 is a nickel-base superalloy with Mo and Co as strengthening elements. It is widely used to manufacture workpieces in high temperature service environment because of its high temperature strength and good corrosion resistance[1–4]. It has been the pursuit of researchers to control the microstructure of metal materials by new methods and technologies, so as to improve the comprehensive properties. In many cases, it is necessary to control the microstructure of the metal workpiece without changing its shape. In the past few decades, it mainly includes heat treatment, carburizing, nitriding, and subzero treatment[5–10]. Compared with other treatment technologies, subzero treatment has the advantages of simple operation, no pollution and low cost[11–12].

The mechanical properties of materials under subzero conditions are quite different from those at room temperature, which are determined by the special microstructure under subzero conditions. In general, workpieces that have undergone subzero treatment have a microstructure with finer grains and strengthening phases[13]. A large number of studies have been reported to improve the microstructure of alloys by subzero treatment, such as aluminum alloys[14], copper alloys[15], titanium alloys[16] and special steel[17] materials. However, there are few studies on the microstructure evolution of nickel-based superalloys during subzero treatment.

Titanium alloys can produce higher dislocation density and promote the formation of twins after subzero treatment[18]. In addition, the grain orientation of titanium alloy changes from (110) to (200) after subzero treatment[19], indicating that the grain orientation and texture composition change during subzero treatment. A large number of studies on steel have shown that after subzero treatment, its hardness and wear resistance can be significantly improved. Jurči et al[20] found that subzero treatment of tool steels refines the size of the martensite region, enhances the dislocation density, and introduces more small spherical carbides than conventional heat treatment. The steel has different microstructures at different subzero treatment temperatures, and the critical temperature of precipitation phase change is about −140 °C[21]. Yan et al[22] carried out different temperature treatments before

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tempering for high speed steel, and proved that the mechanical properties and wear resistance of subzero treated specimens are better than those of untreated specimens. Sobotova et al[23] reported that subzero treatment will produce a large number of substructures and precipitates, which will affect the mechanical properties and wear resistance; this concept was latterly accepted by a large number of researchers. Based on this, the study on microstructure evolution of Inconel 617 alloy by subzero treatment may help to improve the properties of the alloy, thus expanding the application field of nickel-based superalloys.

The microstructure characteristics of Inconel 617 alloy under different subzero treatment time and numbers were studied. X-ray diffraction (XRD), electron backscatter diffraction (EBSD) and transmission electron microscope (TEM) techniques were used to analyze the evolution of grain morphology, dislocation density, texture and lattice constant during subzero treatment. This study provides a preliminary exploration and theoretical guidance for the microstructure control of nickel-based superalloys in subzero treatment.

1 Experiment

The nominal chemical composition of commercial Inconel 617 alloy was Cr20.8, Mo9.12, Al1.28, Co13.1, Fe0.8, C0.07, Si0.11, Ti0.22 and balance Ni. Squares of 20 mm×20 mm×10 mm were cut from as-forged Inconel 617 alloy sheets by wire electrical discharge machining for subzero treatment tests. Fig. 1 shows the original metallographic microstructure of Inconel 617 alloy, which is composed of equiaxed grains (10−100 μm) and annealed twins (indicated by arrow in the figure). The samples were immersed in liquid nitrogen (−196 °C) for different subzero treatment time and treatment numbers. The specific scheme was untreated (origin), once every 12 h (12-1), once every 24 h (24-1), and twice every 24 h (24-2).

After subzero treatment, the samples were ground and polished for the observation by XRD and EBSD. The crystal structure of the sample was analyzed by D/max-2400 X-ray diffractometer, and the target material was Cu (the incident wavelength λ was 0. 154 06 nm), the tube voltage was 40 kV, the scanning range was 10°−90°, and the scanning speed was 2°/min. The TEM sample was first cut into slices with 200 μm in thickness, then polished to 50 μm in thickness by sandpaper, and finally processed into disks with a diameter of 3 mm by Gatan 625 crater instrument. EBSD and TEM specimens were electropolished and double-jet thinned in a mixed solution of 90 mL C_2H_5OH+10 mL $HClO_4$, respectively. The difference is that the polishing voltage was 50 V and the temperature was −20 °C, the thinning voltage was 22 V and the temperature was 0 °C. EBSD data was processed by Channel 5 software.

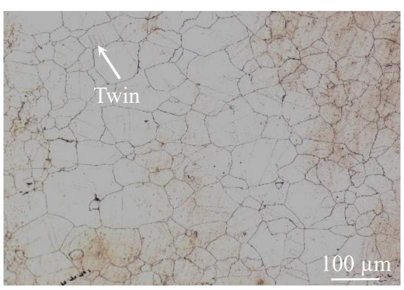

Fig.1 Original metallographic microstructure of Inconel 617 alloy

2 Results and Discussion

The microstructure of the alloy will change during subzero treatment, including lattice constant, grain morphology, texture composition, dislocation density, etc, which will be analyzed in detail below.

2.1 XRD analysis

Fig. 2 is the XRD patterns of Inconel 617 alloy under different subzero treatment conditions. It can be seen that the XRD peak mainly consists of five crystal planes: (111), (200), (220), (311) and (222). There is no new diffraction peak after subzero treatment, which indicates that there is no new phase. However, it is worth noting that the diffraction peak size is different under different conditions. With the increase in subzero treatment time and numbers, the (111) diffraction peak area gradually decreases, as shown in Fig. 2b. At the same time, it can be seen that with the increase in subzero treatment time, the diffraction peak gradually shifts to the right. But after subzero treatment for 24 h, the diffraction peak shifts to the left again. The change of diffraction peak indicates the change of diffraction angle. According to Bragg equation, the larger the diffraction angle, the smaller the crystal plane spacing, and the larger the lattice constant.

In order to better analyze the effect of subzero treatment on the crystal structure of Inconel 617 alloy, the diffraction angle corresponding to the (111) diffraction peak is given in Table 1, and the lattice constant is calculated according to the crystal plane spacing. The lattice constants of the original, 12-1, 24-1, and 24-2 samples are found to be 0.3588, 0.3593, 0.3596, and 0.3594 nm, respectively, and the overall lattice constant is in the range of ± 0.0004 nm. The change of lattice constant and the shift of diffraction peaks are due to the change of stress state and microstructure of the sample after subzero treatment[24]. Subzero treatment will increase the degree of lattice distortion of the matrix, resulting in thermodynamic instability.

2.2 Grain change

Fig.3 shows the inverse pole figures of Inconel 617 samples under different subzero treatment conditions. Black represents high-angle grain boundaries (>15°), and different colors of grains represent different orientations. It can be seen from Fig.3a that a large number of grains in the original sample are in the <111> direction. After subzero treatment for 12 h (Fig.3b), fine grains appear at the high-angle grain boundaries of the original grains, and the fine grains have the characteristics similar to the necklace structure. In addition, it can be seen that the original grain orientation changes from <111> to <101> preferred orientation after subzero

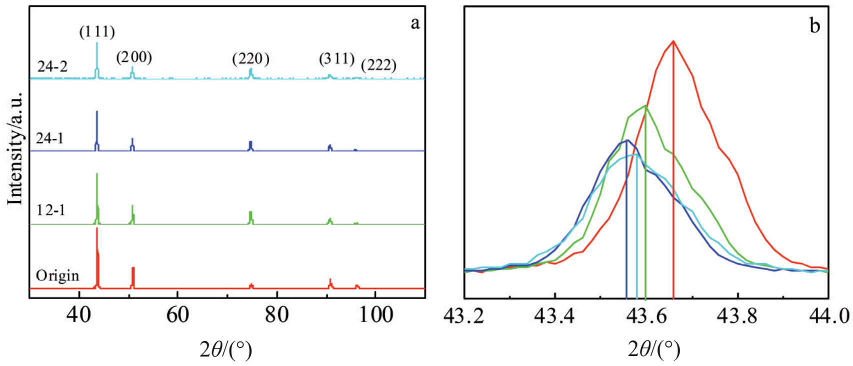

Fig.2 XRD patterns of Inconel 617 alloy under different subzero treatment conditions (a) and locally magnified diffraction peak of (111) crystal plane (b)

Table 1 Diffraction angle (θ) and lattice constant (a) corresponding to (111) peak of the samples

Sample	2θ/(°)	a/nm
Origin	43.660	0.3588
12-1	43.597	0.3593
24-1	43.558	0.3596
24-2	43.578	0.3594

treatment, indicating that subzero treatment can effectively change the crystal orientation of nickel-based superalloys. With the increase in subzero treatment time (Fig. 3c), the number of original large grains gradually decreases and the number of fine equiaxed grains increases. Fig. 3d shows the microstructure of Inconel 617 alloy after two cycles of subzero treatment for 24 h. It can be found that the number of fine grains decreases and the grains grow up after cyclic subzero treatment. The results show that the reasonable subzero treatment time is helpful to refine the grain size, and the cyclic subzero treatment leads to the grain growth.

Fig. 4 shows the average grain size of the samples under different subzero treatment conditions calculated from the EBSD data. In general, the grain size of Inconel 617 alloy can be refined by subzero treatment, but it is affected by the subzero treatment parameters. With the increase in subzero treatment time, the average grain size decreases gradually, and the smallest grain size of 24-1 sample is 26 μm. The original grain refinement may be caused by the change of austenite lattice constant and the fragmentation of the original coarse grains.

2.3 Dislocation

Fig. 5 shows the local misorientation maps of Inconel 617 alloy samples under different subzero treatment conditions. The magnitude of the local misorientation can indicate the

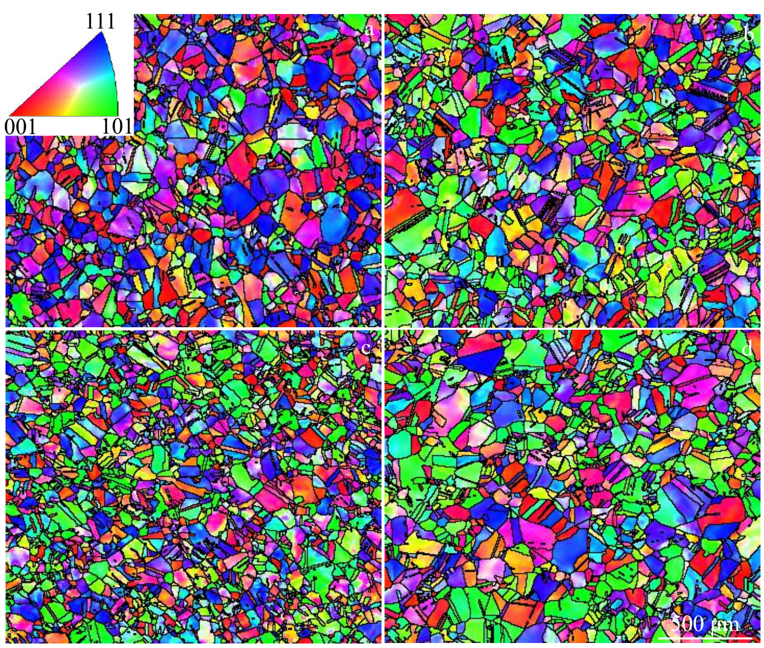

Fig.3 Inverse pole figures of samples under different subzero conditions: (a) origin, (b) 12-1, (c) 24-1, and (d) 24-2

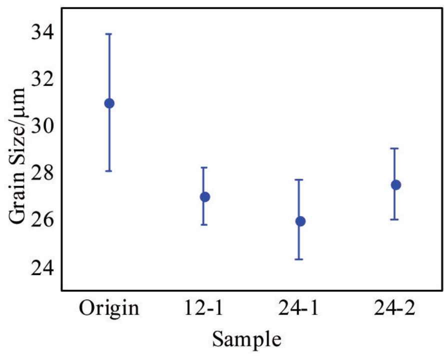

Fig. 4 Effect of subzero treatment on average grain size of the samples

stress value of the material. It can be seen that there is a higher stress at the high angle grain boundaries in the original sample (Fig. 5a), because the grain boundaries can hinder the movement of dislocations. The Inconel 617 alloy after subzero treatment has a high stress around the fine grains (Fig.5a and 5b), which have a high energy relative to the original grains. Because the subzero treatment process is equivalent to a quenching stage, a large number of dislocations are produced at the same time of grain formation, which leads to this phenomenon. After two cycles of subzero treatment for 24 h, the stress of the sample is released, showing a non-uniform stress state.

According to the dislocation theory in continuous media, the dislocations generated in order to coordinate different

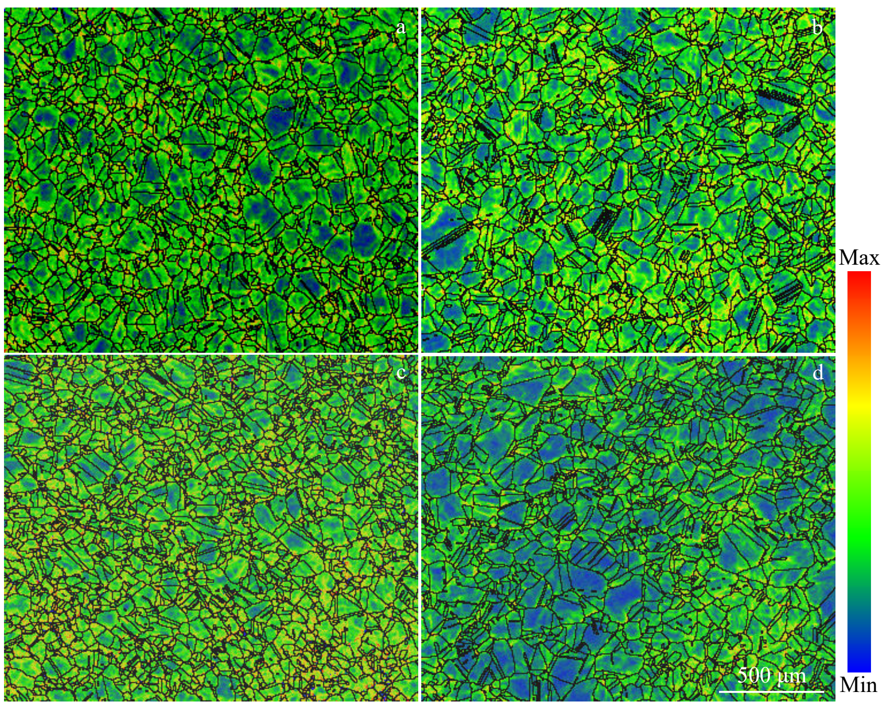

Fig.5 Local misorientation maps of the samples under different subzero conditions: (a) original, (b) 12-1, (c) 24-1, and (d) 24-2

strains of polycrystals are called geometrically necessary dislocations (GND)[25]. In order to better study the dislocation density of the sample during subzero treatment, the GND density of Inconel 617 alloy was calculated. The correlation between GND and local misorientation can be quantitatively analyzed by the following formula [25]:

$$\rho^{\mathrm{GND}} = \frac{2\theta}{\mu b} \tag{1}$$

where ρ^{GND} is the GND density, μ is the unit length of a circle around a feature (that is, the scanning step size, 600 nm), b is the size of the Burst vector (0.25 nm in this study), and θ is obtained by average misorientation. The obtain GND density is shown in Fig.6. After subzero treatment for 12 h, the GND density decreases slightly compared with the original sample. After subzero treatment for 24 h, the density of GND increases significantly to 3.01×10^{14} m^{-2}. However, after twice subzero treatment every 24 h, the density of GND decreases significantly. It can be seen from Fig. 4 and Table 1 that the GND density and average grain size of Inconel 617 alloy do not change directly during subzero treatment.

It can be seen from Fig. 7a that precipitates with different sizes appear, and a large number of dislocation lines are gathered around the precipitates, which are crisscrossed and pile-up. Similarly, after twice subzero treatment every 24 h, precipitates also appear, and the size of precipitates is relatively uniform. By comparison, it can be seen that the number of dislocation lines decreases after twice subzero treatment every 24 h, and the hindrance effect of precipitates on dislocations decreases. The precipitates in the subzero treated samples are found to be simple and complex MC, M_6C and $M_{23}C_6$ carbides with face-centered cubic structure

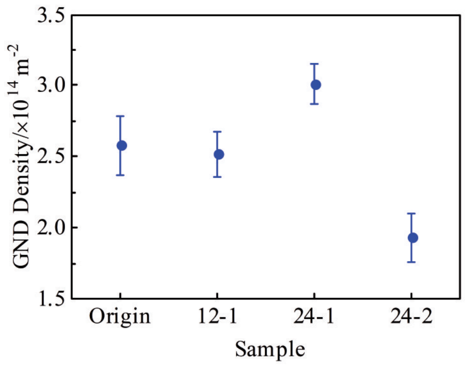

Fig.6 Effect of subzero treatment on average GND density

according to SAED analysis. In the process of subzero treatment, the existence of supersaturated vacancies and other defects make the diffusion coefficient of carbon atoms increase, and the interaction between interstitial carbon atoms and dislocation stress field makes carbon atoms segregate near dislocation lines. During the subsequent recovery treatment to room temperature, the carbon atoms and dislocation lines attract each other, and the carbon atoms near the dislocations diffuse along the dislocation lines and grain boundaries, resulting in the precipitation of carbides. The precipitation of carbides will seriously hinder the movement of dislocations during subzero treatment, thus improving the strength of the alloy. However, the carbides themselves reduce the mechanical properties of nickel-base superalloys, so the effect of carbide precipitation on the properties of Inconel 617 is complex.

2.4 Texture evolution

It can be seen from the inverse pole figure of Inconel 617 alloy shown in Fig. 3 that the preferred orientation of grains has changed after subzero treatment. Therefore, the texture composition and evolution law during subzero treatment are studied in detail in this section. Fig. 8 shows the orientation distribution function of the specimen for the sections of φ_2=0°, 45°, 65° in Euler space. The basic position of the nominal texture component in fcc metals is shown in Fig. 8e. The texture composition of the material can be determined by comparing with the test results. As can be seen from Fig. 8a, the original Inconel 617 sample mainly has rotated cube texture {001}<110> and rotated copper texture {112}<110>. After a single subzero treatment for 12 h (Fig.8b), the rotated cube texture and rotated copper texture are weakened and transformed into brass texture {110} <112> and P texture {110} <122>. After subzero treatment for 24 h, there are not only brass texture and P texture, but also goss texture {011} <001> (Fig. 8c). The brass texture and P texture disappear and only the goss texture is observed in the sample after twice 24 h subzero cycles (Fig.8d).

Fig. 9 shows quantitative analysis of microtexture changes on α-fiber, τ-fiber and γ-fiber of Inconel 617 alloy in Euler space under different subzero treatment conditions. The variation of orientation distribution function values shows that the intensity of the goss texture, brass texture and copper texture of the subzero treated sample is higher than those of the original sample, and the intensity increases with the increase in subzero time, which reaches the maximum after

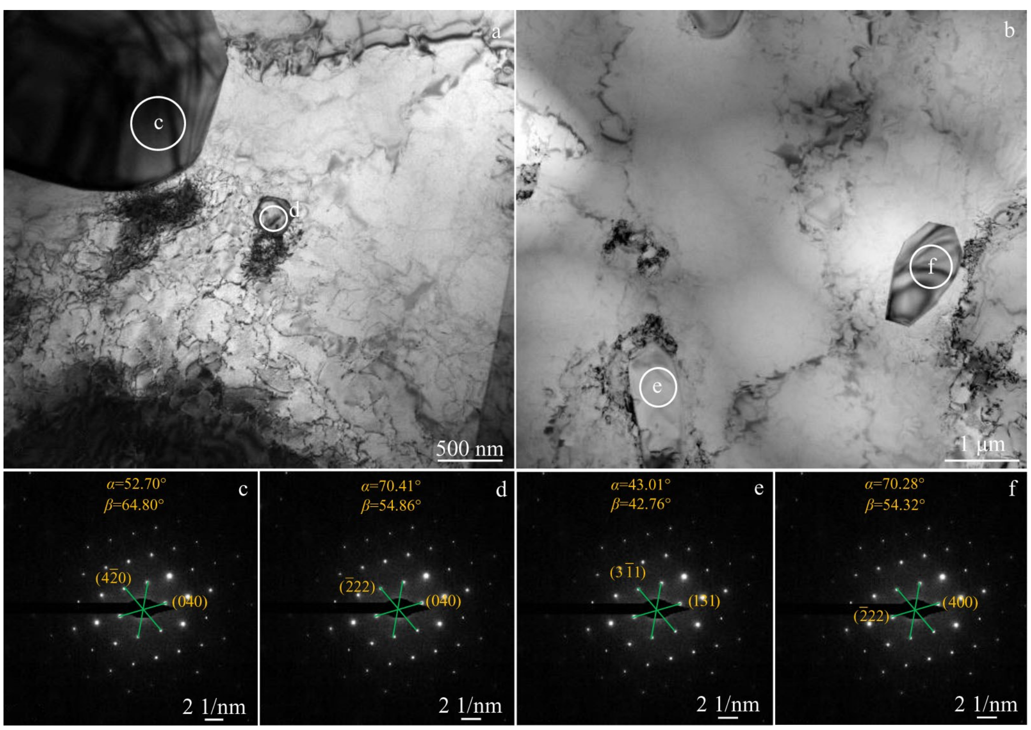

Fig.7 TEM bright-field images of the samples after subzero treatment once every 24 h (a) and twice every 24 h (b) and corresponding SAED patterns of white circle precipitated phases (c−f)

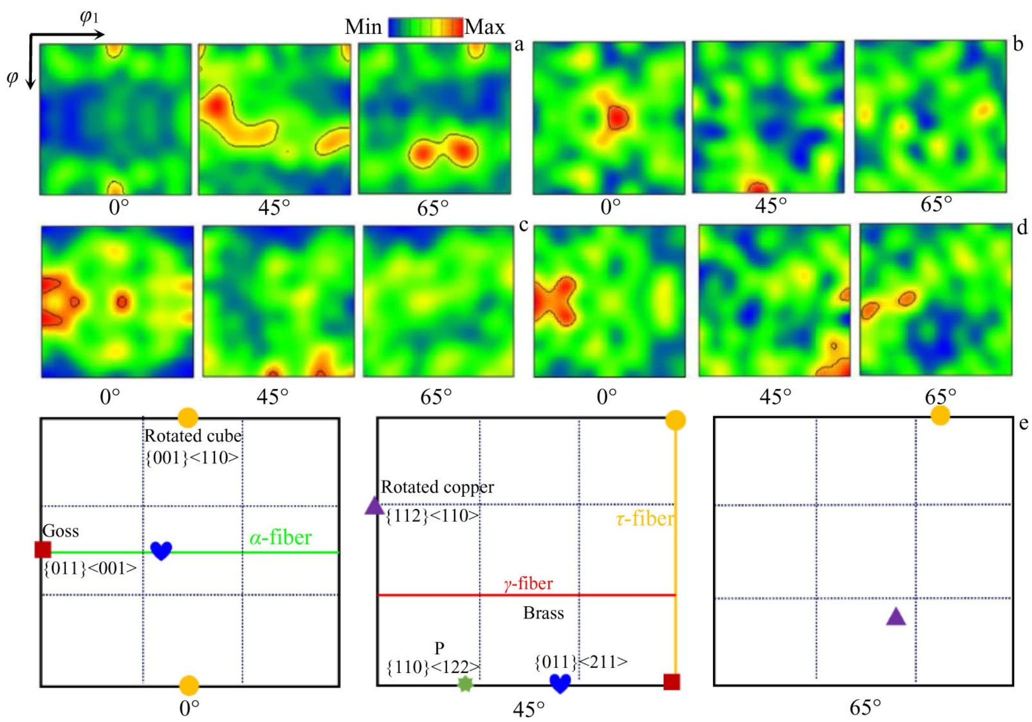

Fig.8 Texture features on different sections in Euler space of the samples under different subzero conditions: (a) origin, (b) 12-1, (c) 24-1, (d) 24-2; basic positions of nominal textures in fcc metals (e)

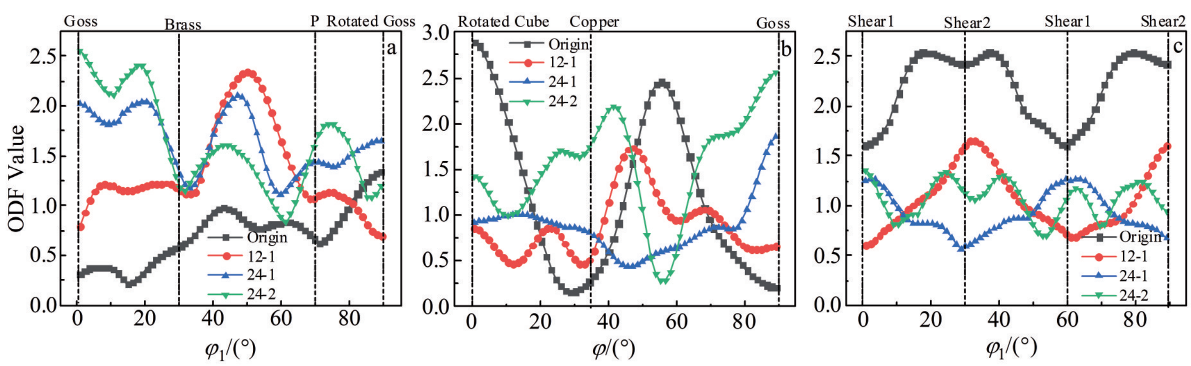

Fig.9 Variation of orientation distribution function values along special orientation lines after subzero treatment of Inconel 617: (a) α-fiber, (b) τ-fiber, and (c) γ-fiber

two cycles of 24 h subzero treatment. It is well known that the composition and type of texture have a great influence on the properties of alloys. To sum up, subzero treatment can effectively control the microstructure of Inconel 617 alloy.

3 Conclusions

1) The grain size is refined and the lattice constant increases after subzero treatment for Inconel 617 alloy.

2) A large number of simple and complex carbides (MC, M_6C and M_{23}C$_6$) are precipitated in the specimen after subzero treatment. The GND density increases after subzero treatment for 24 h, while the GND density decreases after twice subzero treatment every 24 h.

3) With the increase in subzero treatment time, the texture of Inconel 617 alloy transforms from the original rotated cube texture and rotated copper texture to brass texture, P texture and goss texture. With the increase in subzero treatment numbers, the ideal texture component mainly transforms into goss texture.

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深冷处理过程中Inconel 617合金的微观组织演变

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摘　要：研究了Inconel 617合金在不同深冷处理时间和次数条件下微观组织的演变规律。结果表明，深冷处理对Inconel 617合金的微观组织结构有很大的影响，随着深冷处理时间的延长，晶粒尺寸减小，但是随着深冷处理次数的增加，晶粒尺寸又增大，并且细化晶粒的晶界处具有高的应力。晶格常数与晶粒尺寸的变化相反。深冷处理试样析出了MC、M_6C、M_{23}C$_6$的简单和复杂碳化物，导致了位错的塞积。深冷处理24 h后试样的几何必须位错（GND）密度增大，当24 h循环处理2次后，GND密度显著降低。此外，深冷处理导致Inconel 617合金的旋转立方织构和旋转铜织构转变为黄铜织构、P织构、高斯织构。

关键词：Inconel 617合金；深冷处理；晶粒细化；几何必须位错；微观组织

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