

A method for enhancing ductility of a polycrystalline Ni-based superalloy K417G

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Abstract: In this work, the effects of hot isostatic pressing and heat treatment (solution and double aging) on the high-temperature tensile properties of a nickel-based polycrystalline superalloy K417G were investigated. The experimental results indicate that following the hot isostatic pressing and heat treatment, the porosity of the alloy decreases from 0.072% (in as-cast state) to 0.043%. The volume fraction of γ' phase increases from 43.28% to 56.54%, and the shape tends to be more cubic. The plasticity of the superalloy at 900 °C increases from 5.1% in as-cast state to 9.8% followed HIP and HT, but the tensile strength remains unchanged. Large size micropores are present in the as-cast sample and cracks sprout and expand at the micropores. After hot isostatic pressing and heat treatment, micropores are effectively eliminated, preventing them from becoming sources of cracks. This delay in the emergence of cracks results in enhanced plasticity. In addition, dislocations in the specimens after hot isostatic pressing and heat treatment bypasses the γ' phase through the Orowan mechanism, leading to a further increase in plasticity.

Keywords: hot isostatic pressing; heat treatment; microstructure; fracture model; mechanical properties

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1 Introduction

Since the mid-20th century, cast superalloys have been rapidly developed in the aerospace field, and they are mainly used in hot-end components of aeroengines, such as turbine blades, turbine disks, guide vanes, and combustion chambers^[1-3]. K417G superalloys not only have the advantages of low density, good plasticity, and high medium-temperature strength, but also are inexpensive and have good stability. The as-cast structure of the alloy consists of the γ phase as the matrix, within which the ($\gamma+\gamma'$) eutectic and carbides are uniformly dispersed. Additionally, numerous γ' strengthening phases are present within this microstructure^[4, 5]. With an increase in the size of as-cast K417G superalloy

components, the microscopic porosities and shrinkage cavities caused by solidification and shrinkage increase, thereby increasing the starting points for crack initiation and greatly reduces the performance of the parts^[6-8]. In addition, the smaller volume fraction and coarser shape of the γ' phase in cast superalloys weaken the strengthening effect of the γ' phase and has an adverse effect on the mechanical properties.

Hot isostatic pressing (HIP) technology, which combines high-temperature and isostatic pressing, is an effective method for eliminating microscopic holes and loose defects inside castings. Through the combined action of high temperature and high pressure, the castings are subjected to the same pressure to achieve densification^[9-11]. Roncery et al.^[12] reported that appropriate HIP and heat treatment can effectively reduce the number of micropores in a material and refine the γ' phase in an alloy, thereby improving its durable creep. Zheng et al.^[10] reported that micropores in investment castings are usually located at the solidification front of the γ/γ' eutectics, with sizes ranging from a few microns to tens of microns. Micropores are closed through the coalescence of fine γ' phase during HIP, and the diffusion of solute into the porosities induces the formation of γ' phase. However, the slow

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cooling rate of the HIP treatment coarsens the γ' phase, so that the performance of the casting is reduced. Therefore, the alloy needs to be heat-treated to readjust its structure and improve its performance^[13-15]. He et al.^[16] conducted appropriate heat treatment on the third-generation single-crystal superalloy DD33 after HIP. The size and volume fraction of γ' phase can be basically the same as those of the standard heat-treated alloy. The volume fraction and equivalent diameter of the micropores were significantly reduced, which resulted in a significant increase in the persistence performance compared to that of the standard heat-treated state. Sentyurina et al.^[17] used HIP and heat treatment processes to comprehensively treat a sample, increasing the tensile strength, yield strength, and elongation of the alloy by 31%, 21%, and 62%, respectively. Yuan et al.^[18] eliminated more than 80% of the shrinkage cavities in K452 superalloys after HIP, and the diameter of the residual shrinkage cavities decreased to below 10 μm . However, the HIP temperature is proximate to the dissolution temperature of the γ' phase. This proximity results in partial dissolution of the γ' phase into the matrix. Subsequent slow cooling induces re-precipitation and growth of the γ' phase, leading to pronounced coarsening. This coarsening of the γ' phase adversely affects the endurance life of the alloy. On this basis, solution and double aging heat treatment was carried out to restore the degraded structure of K452 via HIP. The durability life was restored and greater than that of the sample without HIP.

To date, the influencing mechanisms of the combined HIP and solution and double aging heat treatment (HT) method on the microstructures and properties of polycrystalline superalloys remain unclear. Therefore, in this study, the K417G superalloy was used as the research object, the microstructures of as-cast, HIP, and HIP+HT samples were investigated and compared, the differences in mechanical properties and fracture modes among the various samples were also examined. This study provides theoretical basis for HIP and HT of K417G superalloy.

2 Experimental procedure

Commercial K417G superalloy was used in this experiment. The chemical composition of the K417G superalloy is listed in Table 1.

Table 1: Chemical composition of K417G superalloy (wt.%)

Ni	B	Al	C	Mo	V	Zr	Cr	Ti	Co
Bal.	0.017	5.23	0.17	3.04	0.75	0.063	8.92	4.3	10.18

To control the defects of K417G, HIP was used with the following parameters: pressure of 170 MPa, temperature of 1,185 $^{\circ}\text{C}$, and duration of 4 h. Then, the sample was cooled at a rate of 8–10 $^{\circ}\text{C}\cdot\text{min}^{-1}$. To further optimize the size and distribution of the γ' phase and improve the overall performance of K417G, the samples were subjected to solid solution treatment at 1,220 $^{\circ}\text{C}$ for 4 h and at 1,080 $^{\circ}\text{C}$ for another 4 h, followed by air cooling and aging at 980 $^{\circ}\text{C}$ for 16 h. Tensile samples were prepared according to ASTM E21 standards

and subjected to 900 $^{\circ}\text{C}$ tensile testing on an universal testing machine. The dimensions of the test samples are shown in Fig. 1. An Olympus BX51M optical microscope (OM), an FEI (Quanta 450 FEG) field emission scanning electron microscope (FE-SEM) equipped with energy-dispersive X-ray spectroscopy (EDS), and a JEOL (JEM 2100F) transmission electron microscope (FE-TEM) with EDS (Oxford X-MaxN) were used to evaluate the microstructures. To observe the microstructures via OM and SEM, samples were initially prepared with P60 to P3000 abrasive papers followed by a 1.5 μm diamond suspension for polishing. Then, the solution, consisting of 20 g of CuSO_4 , 100 mL of HCl, 5 mL of H_2SO_4 and 100 mL of H_2O , was used to reveal the microstructures. Additionally, the samples for TEM investigation were ground down to about 60 μm and punched into round pieces with 3 mm in diameter. Afterward, TEM foils were thinned by twin-jet electropolish in a solution consisting of 10% perchloric acid and 90% alcohol at -25°C and a voltage of 21 V.

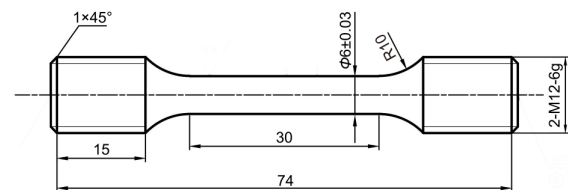


Fig. 1: Sizes of 900 $^{\circ}\text{C}$ tensile test K417G sample (unit: mm)

3 Results

3.1 Microstructure

Figures 2(a, b, and c) show the pore distributions of the alloy in the three states. Micropores are clearly present in the as-cast samples, while they are eliminated in both the HIP and HIP+HT samples. Figures 2(d, e, and f) show SEM images of the samples. In all the three states, the alloy is composed of the γ matrix phase, γ' strengthening phase, γ/γ' eutectic, and carbides. The carbides in the as-cast and HIP samples are mainly in the form of blocks and strips, while those in the HIP+HT sample are mainly chain-like. In all the three states, the carbides are mainly distributed at the grain boundaries and between dendrites. EDS results of the samples indicate that the alloy mainly contains Mo, Ti, and Cr, and possibly MC, M_6C , and M_{23}C_6 , which require further testing and analysis. Figures 3(g, h, and i) show diagrams of the morphologies and distributions of the γ' phase. The γ' phase of the as-cast sample exhibits an irregular shape, mainly irregular polyhedrons, and its size is concentrated at 0.3 μm . The γ' phase of the HIP sample also exhibits an irregular shape. The phase consists of irregular large concave bodies and small polyhedrons. Statistics show that the size of the γ' phase is concentrated below 0.3 μm . The γ' phase in the HIP+HT sample shows an ordered and neatly arranged cubic structure. Compared with those of the as-cast and HIP samples, the size distribution of the HIP+HT sample is more concentrated at 0.3 μm . This indicates a reduced presence of the γ' phase in both smaller and larger size ranges

compared to the other two samples. Additionally, the γ' phase exhibits greater uniformity in size distribution.

Figure 3(a) shows the changes in porosity and maximum pore size. The porosities in the as-cast, HIP, and HIP+HT states are 0.072%, 0.034%, and 0.043%, respectively. Micropores are significantly reduced in the HIP samples compared to the as-cast samples. The porosity decreases by 52.78%, and the maximum pore size decreases from 236.54 μm to 34.33 μm . In addition, after heat treatment, the porosity does not increase significantly, still remains 40.28% lower than that of the as-cast sample. Measurements of the volume fractions and average sizes of the γ' phases in the three sample states show that the corresponding volume fractions are 43.28%, 40.64%, and 56.54% for the as-cast, HIP, and HIP+HT samples, respectively, as shown

in Fig. 3(b). After HIP+HT, volume fraction of the γ' phase increases by 13.26% compared to that of the as-cast sample. The average sizes of the γ' phase in the as-cast, HIP, and HIP+HT samples are 0.41 μm , 0.39 μm , and 0.33 μm , respectively.

3.2 Mechanical properties

The tensile engineering stress-strain curves of the as-cast, HIP, and HIP+HT samples at 900 $^{\circ}\text{C}$ are shown in Fig. 4(a). The yield strength and tensile strength of the samples under the three conditions are almost the same. The elongation of the as-cast sample is 5.1%, while that of the HIP sample is 8.7%, which is 70.59% greater than that of the as-cast sample. The elongation further increases to 9.8% after HIP+HT. The work hardening curves of the alloy in the three states are shown in Fig. 4(b). In

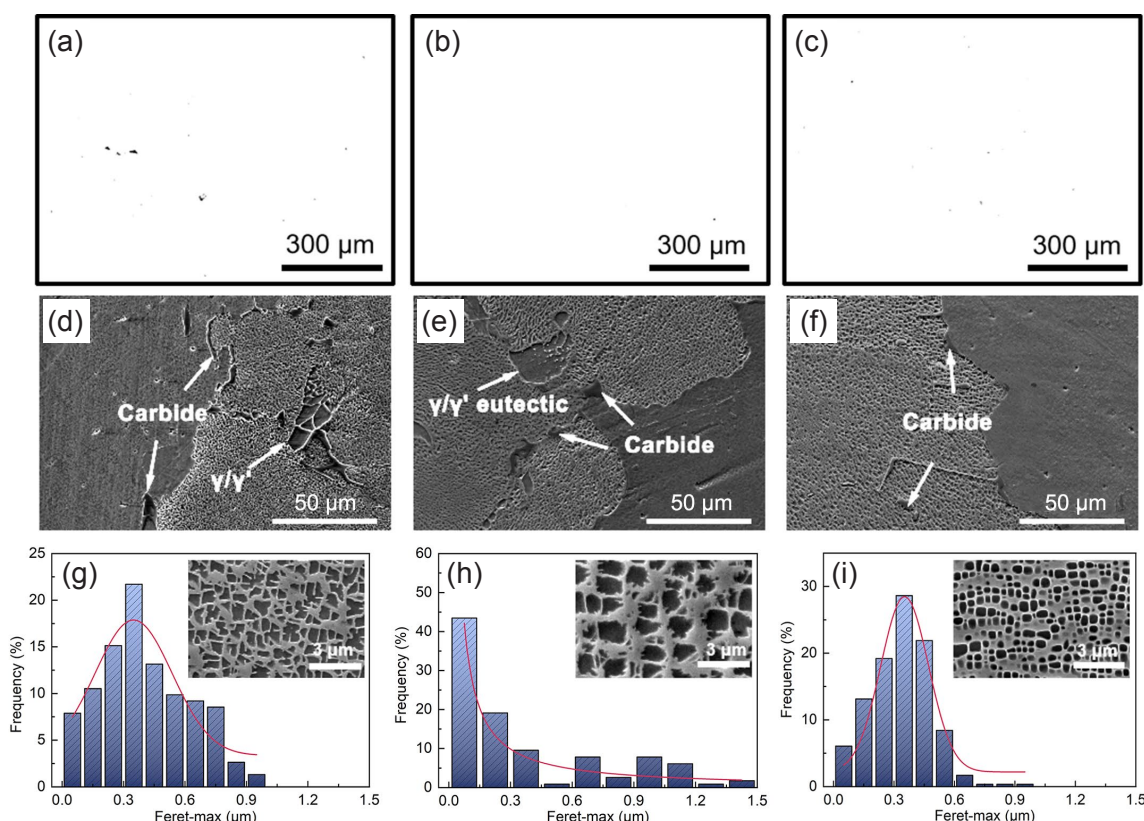


Fig. 2: Pore distributions (a, b, c), microscopic morphologies (d, e, f), and SEM images and size distributions of γ' phases (g, h, i) in the three states: (a, d, g) as-cast; (b, e, h) HIP; and (c, f, i) HIP+HT

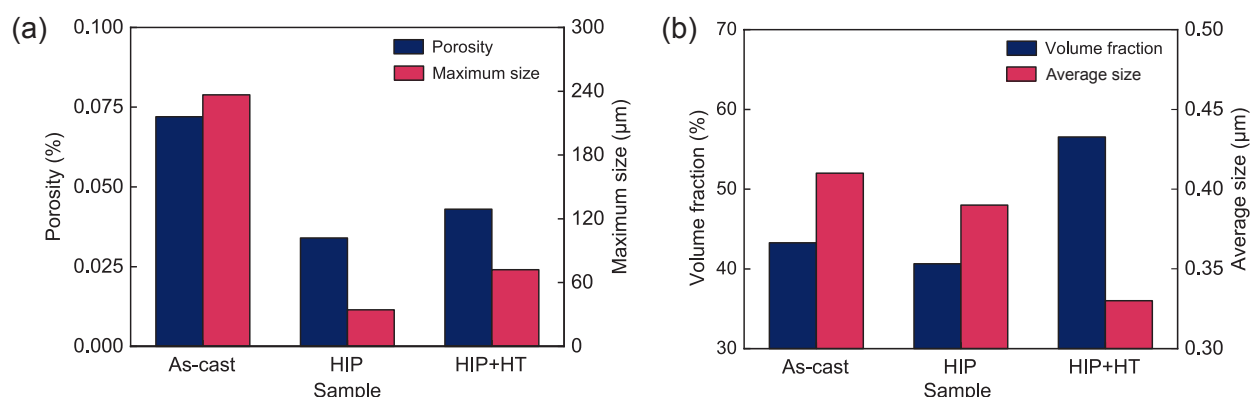


Fig. 3: Changes in porosities and maximum sizes of micropores (a), and volume fractions and average sizes of γ' phases in the three samples (b)

the three states, the work hardening rate of the alloy decreases with increasing strain. The rate of decrease in the as-cast sample is the fastest, and the HIP+HT samples show the lowest decreasing rate in strain hardening rate. The strain hardening rate of the HIP+HT samples is consistently higher than that of the as-cast and HIP samples, demonstrating that HIP+HT has a positive impact on the load-bearing capacity of the alloy during the work hardening stage.

3.3 Fracture morphology

The tensile fracture surfaces of the alloy for the three conditions, tested at 900 °C, are shown in Fig. 5. The fracture surface of the as-cast sample shows an obvious dimple structure, which is a typical ductile fracture mode. Numerous micropores and coalesced cracks are observed near the dimples. This phenomenon occurs because, under external loading, stress

concentrates at the micropores. The stress transfer is hindered in this area, causing the micropores to become regions of stress concentration. This concentration accelerates the formation of cracks at the micropores, turning them into one of the sources of alloy fracture. Figure 5(b) shows the fracture morphology of the HIP samples. The cracks show obvious dendritic shape and exhibit a transgranular fracture mode. As shown in Fig. 5(e), a small number of lumps appear on the fracture surface of the sample, exhibiting a flat and stepped appearance. The cracks are dispersed without any obvious pattern in their distribution. When the stress is blocked between the massive brittle phase and the dendrites, the fracture in this area is accelerated. After the sample undergoes HIP+HT, a small number of micropores appear that are accompanied by cracks. Then, small dimple-like fractures appear in local areas, and many strips adhere to the fracture surface, which is a mixed fracture, as shown in

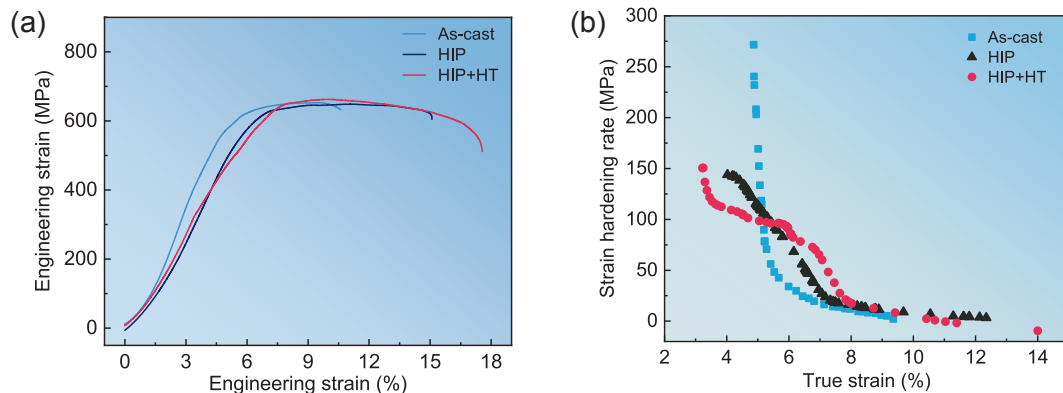


Fig. 4: Engineering stress-strain (a) and work-hardening (b) curves of the three samples

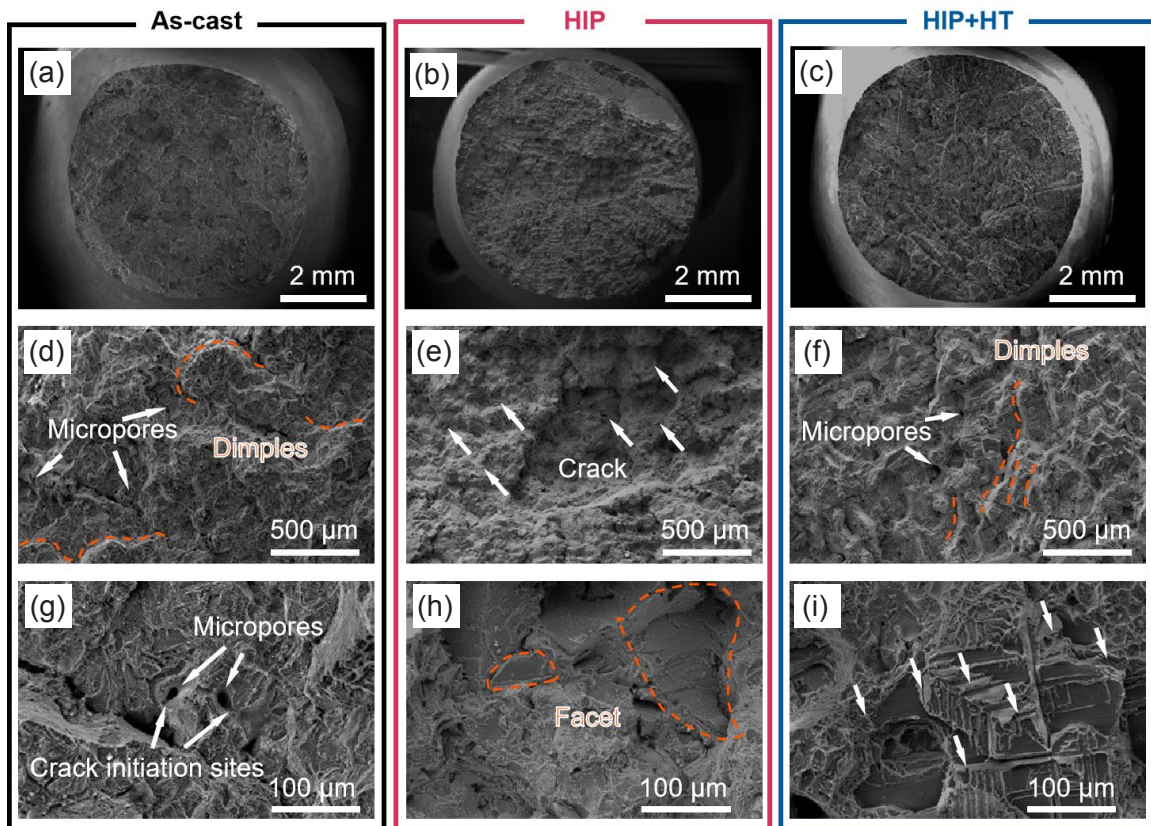


Fig. 5: Tensile fracture morphologies of samples at 900 °C: (a, d, g) as-cast; (b, e, h) HIP; and (c, f, i) HIP+HT

Figs. 5(f and i). These findings indicate that, under the given conditions, regions of solute depletion or microvoids associated with incomplete solid solution formation can emerge in the alloy after heat treatment, acting as preferential sites for crack initiation. Nevertheless, the matrix and precipitate phases are effectively strengthened through the heat treatment, enabling the alloy to absorb significant deformation energy and thereby retard the crack propagation rate.

The longitudinal cross-sectional structures of the fractured tensile samples under the three different conditions were observed, as shown in Fig. 6. The longitudinal section of the fracture in the as-cast sample exhibits non-uniformity, featuring a slope-like region that arises from plastic deformation. Cracks at micropores are observed near the fracture and are connected and expanded with nearby cracks, which is the main fracture

mode in this state. After HIP treatment, the cracks in the tensile sample exhibit transgranular fracture mode. The fracture morphology is relatively flat with almost no plastic deformation. The γ' phase exhibits a rafting phenomenon, and the rafting direction is parallel to the stress direction, as shown in Fig. 6(e). The blocks are debonded near the fracture, indicating that in this state, the mode of tensile fracture is transgranular fracture resulting from carbide fragmentation, matrix debonding, and matrix fracture. After the sample is subjected to HIP+HT, the tensile fracture surface is smooth, with small regional slope-like areas, and some plastic deformation occurs. Near the fracture, the carbides are fragmented in the eutectic region and debonded from the matrix, indicating that under these conditions, the fracture mode is mainly transgranular fracture caused by fragmentation of intragranular carbides.

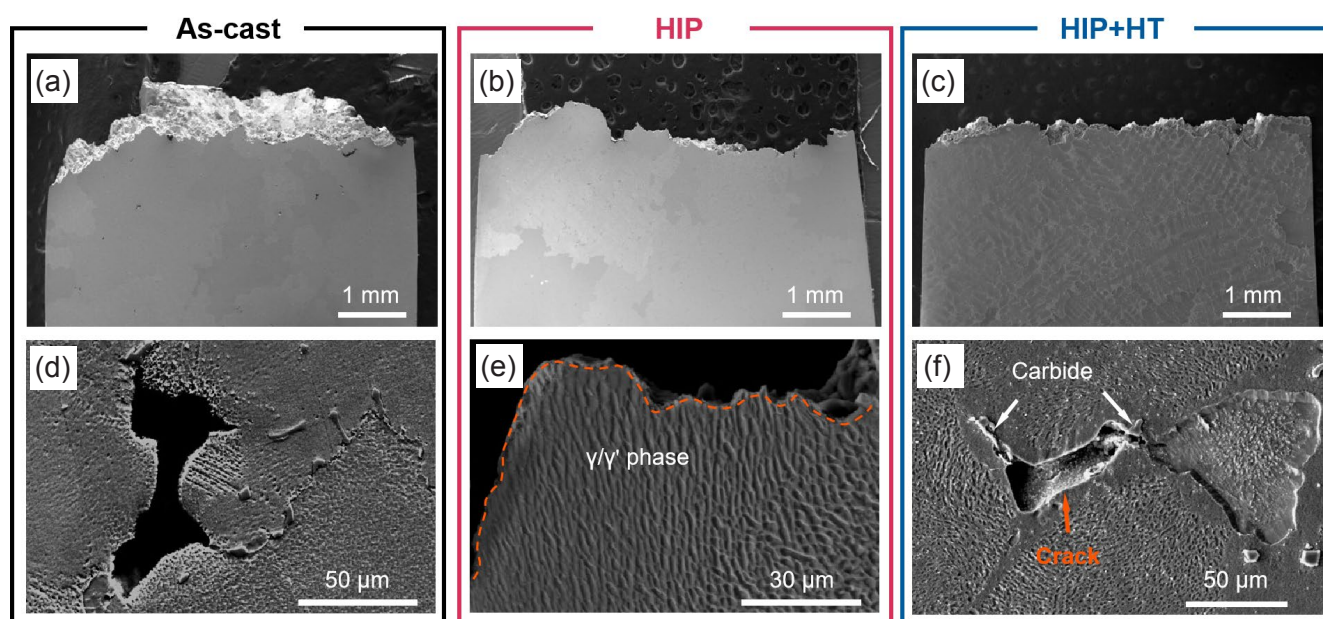


Fig. 6: Longitudinal cross-sectional morphologies of 900 °C tensile fractures of as-cast (a, d), HIP (b, e), and HIP+HT (c, f) samples

4 Discussion

4.1 Effects of micropores on tensile properties of K417G superalloys at 900 °C

As a kind of defect in superalloys, micropores play a crucial role in the fracture mechanism of alloys^[19, 20]. In a stress environment, micropores initially forms on the surface where micropores already exist, as shown in Figs. 5(g) and 6(d). Over time, due to the non-uniform size distribution of micropores and their growth rates, governed by a power-law relationship, some micropores grow at accelerated rates compared to others, resulting in the development of a few exceptionally large micropores through preferential coalescence and localized stress concentration^[21-23]. Studies have shown that this initial micropore coalescence is a relatively stable process and usually does not directly lead to alloy failure or have a significant adverse impact on yield strength^[24]. However, after the alloy has undergone the ductile deformation stage, the micropores

associated with early coalescence begin to undergo a second growth process, which is accompanied by gradually increasing ductile deformation. When micropores grow to a critical size, they can act as nucleation sites for microcracks. The second coalescence stage leads to the phenomenon of strain localization, i.e., nonuniform strain distribution in the alloy, which may lead to the generation of ductile damage^[25]. In addition, considering the characteristics of micropores, especially their large specific surface area and small density, the presence of micropores in superalloys not only affects the toughness of the alloy but also directly affects the crack expansion path and rate and the crack propagation resistance. The relational expression of crack propagation resistance (I) is as follows^[26]:

$$I = D\sigma_0 F(f_0, f_E) \quad (1)$$

where D is the spacing between micropores; σ_0 is the initial tensile yield stress; f_0 is the initial micropore volume fraction; and f_E is the critical point at which micropores coalesce to form

a new crack surface (usually when $f_E \approx 0.2$); F is a function of f_0 and f_E . As f_0 increases, F firstly increases rapidly and then increases slowly^[25-26]. In the as-cast sample, the initial micropore volume fraction f_0 is relatively high, as shown in Fig. 4(a), and the crack propagation resistance (I) required in the fracture process is low according to Eq. (1). This discovery indicates that micropores transform into plastic zones when subjected to a specific tensile force, consequently facilitating their expansion. When a plastic fracture surface is removed and the micropores are in relatively close proximity, some of the micropores may become interconnected during the early growth stages. This interconnection can lead to the formation of dimples. Subsequently, these micropores may separate from the plastic deformation area at the bottom of the dimple before significant plastic deformation occurs, as shown in Fig. 5(g). After HIP, the initial micropore volume fraction f_0 decreases, as shown in Fig. 4(a), resulting in an increase in the distance D between micropores. In this case, the crack propagation resistance (I) rises in accordance with Eq. (1). As a consequence, the number of micropores undergoing ductile tearing diminishes. When these micropores separate, uniform dimples form on the surface, which in turn impedes crack expansion, as shown in Fig. 5(e). In addition, as a result of the decrease in both the quantity and dimensions of micropores within the HIP sample, the stress concentration in the internal micropores is mitigated, causing some micropores to attain $f_E=0.2$ at a later stage. This result makes it difficult for the

existing micropores to coalesce again and form new cracks, thereby improving the tensile plasticity of the sample.

4.2 Effects of microstructures on tensile properties of K417G superalloys at 900 °C

Studies showed that the plasticity of superalloys depends on the local embrittlement of the alloy caused by carbides or segregation, the deformation mechanism affected by local strains, and the instability of the γ' phase at high temperatures^[27, 28]. Fractographic analysis indicates that fracture in the HIP-processed samples initiates preferentially at carbide particles, as shown in Fig. 7, rather than at the micropores. This indicates that in the HIP samples, micropores no longer act as localized regions of strain. Instead, localized strains are generated at the carbides, leading to the development of cracks and subsequent fracture of the alloy. Therefore, the disappearance of micropores in the HIP-treated samples causes a change in the fracture mode and is responsible for the increase in elongation.

In the HIP+HT sample, according to $MC + \gamma \rightarrow M_{23}C_6 + \gamma'$, carbides changed from MC to $M_{23}C_6$ after solid solution and aging heat treatment^[29, 30]. Figure 8 shows the EDS mapping of carbides in HIP+HT samples. It can be seen from Fig. 8 that this carbide is rich in Cr and C, and the content of Mo is less. The dislocation distribution at the fracture surface after tensile fracture is shown in Fig. 9. The dislocation network in the HIP sample is observed to be entangled in the matrix phase, and some dislocations cut through the strengthening phase.

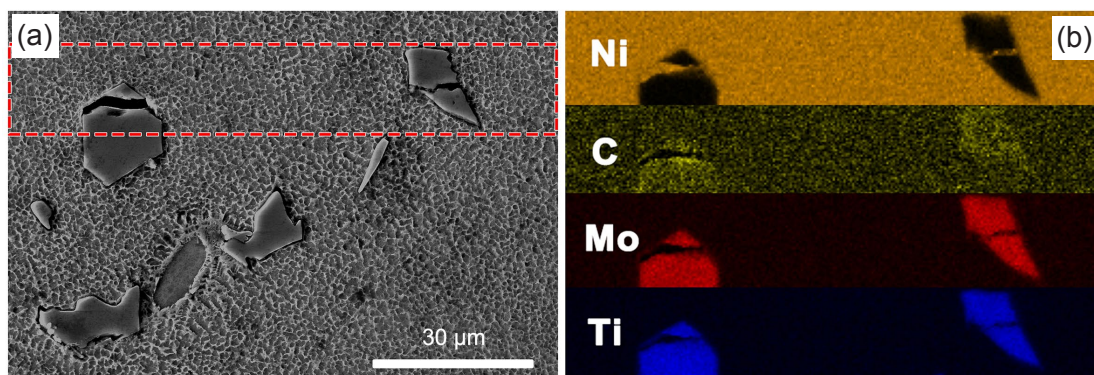


Fig. 7: Morphology of HIP sample near its fracture (a) and SEM-EDS results (b)

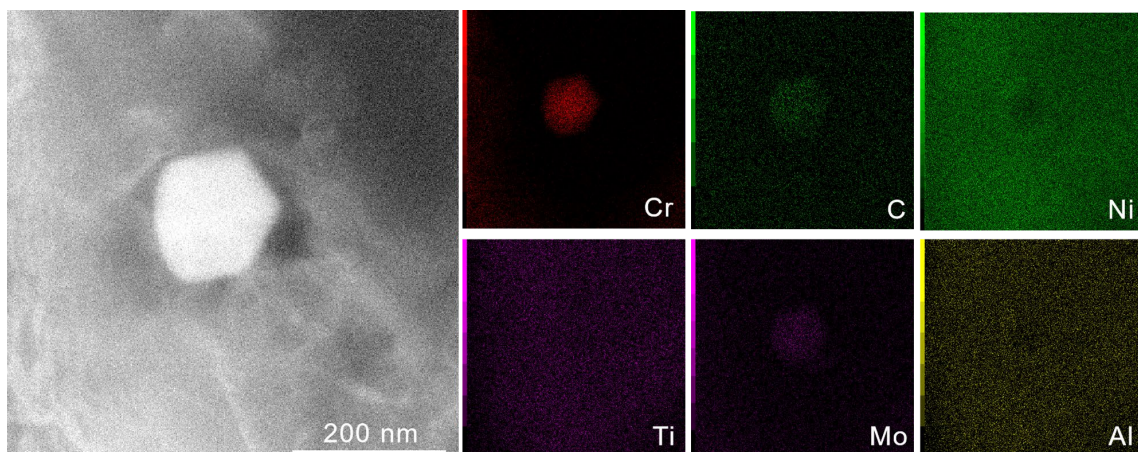


Fig. 8: TEM-EDS results of carbide in HIP+HT sample

As shown in Fig. 9(a), after heat treatment, dislocations are mainly concentrated in the matrix phase. When the dislocation motion encounters the γ' phase, it is pinned and bypasses the γ' phase to form an Orowan ring, as shown in Fig. 9(b). The phenomenon of dislocation entanglement around carbides is observed in the HIP + HT sample, as shown in Fig. 9(c). The selected electron diffraction of carbides indicates that such carbides are $M_{23}C_6$, as shown in Fig. 9(d). Under the action of stress, carbides hinder the movement of dislocations and play a strengthening role. In addition, heat treatment changes the morphology and size of the strengthening phase, thus affecting the migration behavior of dislocations. Studies showed that

after heat treatment, the γ' phase exhibited a highly cubic crystal structure and ordered arrangement, which changed the movement mechanism of dislocations^[31, 32].

Combining the 900 °C tensile properties of the alloy with those of the fracture surface, it can be concluded that the fracture mode of the 900 °C tensile sample under the three conditions primarily features transgranular fracture. Figure 10 shows a schematic diagram of the tensile fracture of the sample under the three conditions. There are many micropores in the as-cast sample. When stress transmission within an alloy encounters micropores, it results in local stress concentration, and these micropores serve as the primary source of cracking,

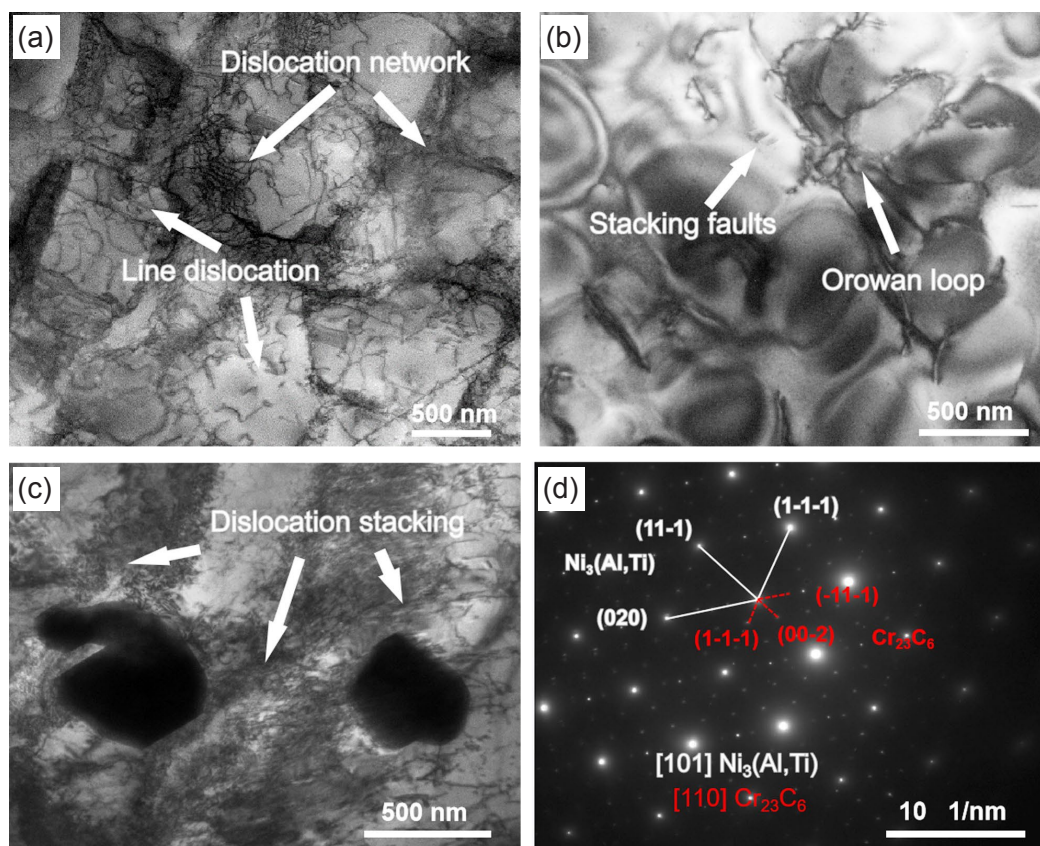


Fig. 9: Dislocation structures (a to c) and corresponding diffraction patterns (d) of the HIP (a) and HIP+HT (b, c, d) samples when stretching at 900 °C

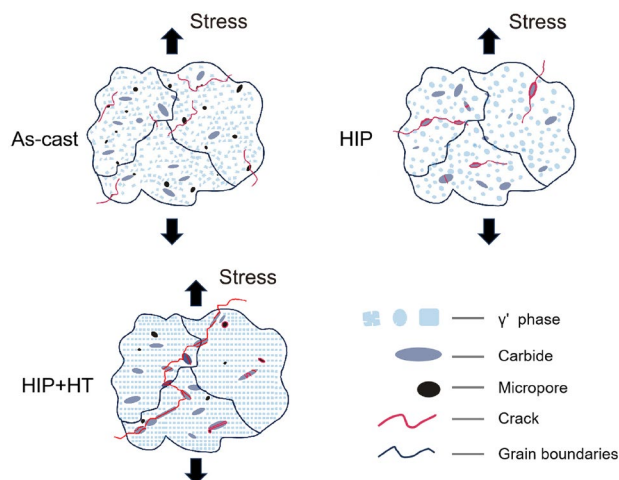


Fig. 10: Schematic diagram of tensile fracture at 900 °C under three conditions

as shown in Figs. 5(g) and 6(d). After HIP, the micropores are no longer the source of cracking. In addition, when stress is transmitted within the alloy during a tensile test conducted at 900 °C, it encounters substantial obstacles of large carbides and coarse, irregular γ' phase precipitates. This encounter increases the internal stress of the materials. Carbides are brittle phase, so cracks will first appear at the carbides, and then extend to the matrix phase. After HIP+HT, the γ' phase attains a stable elemental distribution. During the hardening phase, the alloy's dislocation-carrying capacity is significantly enhanced, with the majority of dislocations being distributed within the matrix, as shown in Fig. 9(b). In this case, when the dislocation density reaches a critical value, due to the exceptional stability of the γ' phase, cracks tend to initiate preferentially at the carbides. As these cracks propagate, the alloy eventually fractures.

5 Conclusions

In this study, as-cast, HIP, and HIP+HT K417G samples were subjected to 900 °C tensile tests. Then, the micropores and microstructures of the samples were investigated to explore the effects of fracture morphology and dislocation changes at fracture on the plasticity of the alloy. Additionally, the tensile fracture modes and plastic changes of the samples in the three states were investigated, and the following conclusions were drawn:

(1) The 900 °C tensile strength of as-cast K417G sample is 665 MPa and the elongation is 5.1%. After hot isostatic pressing and heat treatment, the elongation is increased by 92.16%, while the strength is almost unchanged.

(2) The porosity of the as-cast sample is 0.072%, with a volume fraction of the γ' phase amounting to 43.28%, and an average size of the γ' phase measuring 0.41 μm . Following HIP+HT, the porosity diminishes to 0.043%, the volume fraction of the γ' phase increases to 56.54%, and the average size of the γ' phase decreases to 0.33 μm .

(3) The tensile fracture mode of the as-cast samples at 900 °C is dominated by microvoid coalescence and extensive crack propagation. In both the HIP and HIP+HT samples, carbides serve as the origin of cracks. The onset of crack generation is postponed, which ultimately leads to an enhancement in alloy plasticity. Dislocations in the HIP+HT samples bypass the γ' phase via the Orowan mechanism, resulting in increased alloy plasticity.

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Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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