Nanocrystallization of Ni-based superalloy K403 by laser shock peening

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Abstract: The surface of the K403′s thin slice specimen uses laser shock processing technology in order to strengthen the surface on the nanocrystallization, located on the surface of the specimen, for the cast parts of the nickel-based superalloy K403, which produced cracks, corrosion, and wear. The formation mechanism of the nano-crystal layer on the surface was analyzed by these technologies using X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The results showed that a nanocrystalline layer of 226 nanometers thick could be prepared by laser-induced high-pressure plasma shock wave on the specimen′s surface. The results of XRD and SEM showed that it will not change the phase structure by that laser shock processing the refined surface grain structure. Under the high pressure, the surface microstructure of K403 specimen generated a great number of dislocations and refinement grains into nanoscale.

Key words: LSP; nanocrystallization; TEM; dislocation

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激光冲击强化 K403 镍基高温合金表面纳米化

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摘 要:针对 K403 镍基高温合金铸造构建易发生裂纹、腐蚀、磨损的问题,采用激光冲击强化技术对 K403 薄片试件进行处理,使试件表面纳米化提高材料力学性能。利用 X 射线衍射、SEM 扫描电镜、TEM 透射电镜分析了材料表面纳米晶层的形成机理。实验结果表明:激光诱导的高压等离子体冲击波可以在样品表面上形成 226 nm 厚的纳米晶层;从 SEM 和 TEM 结果可以看出,激光冲击强化不会改变材料表面物相。在高冲击波压力下,K403 试样表面组织产生了位错和纳米级晶粒细化。

关键词:激光冲击强化; 纳米化; 透射电镜; 位错

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0 Introduction

Nickel-based superalloy K403 is a widely used cast, possessing high temperature strength, corrosion and oxidation resistance, and fatigue resistance [1]. It is commonly used to make the high-pressure turbine blade[2]. While the aero-engine is at work, the parts made by K403 can withstand high temperature, vibration, and fatigue. Unfortunately, it will inevitably produce cracks, corrosion and wear, which will limit its service time under harsh working condition. However, wear, corrosion, and cracks remain on the surface behavior of material, it is a effective way to improve the performance of anti-wear, anti-corrosion and anti-fatigue by the appropriate surface treatment improving the superalloy K403′s surface properties[3-4] Without changing the base material, the alloy can get a certain thickness of the nanocrystalline organization depending on the severe plastic deformation on material´s surface, that realized the surface nanocrystallization. The surface of the nanocrystallization has a great application prospect, and many scholars are currently working on the surface of nanocrystallization. Lu etc. adopted surface mechanical attrition treatment(SMAT) to recognize and promote the low nitriding technology. Hanlon[7] etc. adopted ultrasonic shot peening technology (USSP) to prepare a nano-Na on the surface of pure Ni and studied the effect of surface nanocrystallization on the fatigue behavior and the cracks′ expansion. Unfortunately, the existing preparation technology confines the equipment′ s structure or material component is hard to meet the need of processing complex structure components, like high-pressure turbine blades, thereby limiting the application and promotion of the surface nanocrystallization on a nickle-based superalloy.

In recent years, laser shock processing (LSP, showed in Fig.1) adopted short pulse(NS level), high-peak power density (GW/cm^2 level) laser to irradiate the metal material′ s surface which makes the absorption protective layer casting on the surface to absorb energy, as a new surface nanocrystallization strengthening technology. They tend to have an explosive gasification change and then produce the high-pressure plasma shock wave. This shock wave is used directly on the material′ s surface to form the residual compressive stress by changing the microstructure that improves performance of anti-fatigue, anti-corrosion and anti-wear [5]. Now, laser shock processing have been successfully preparing nanocrystalline on the surface of Al, Ti [6-7] and etc. Notably, it is seldom reported that the surface nanocrystallization on nickle-based superalloy is induced by laser shock processing.

This paper adopts laser shock processing in order to administer the shock processing of the Ni-based superalloy K403 thin slice specimen. The X-ray diffraction instrument was used to evaluate the structure and composition of the material specimen, SEM and TEM are used to observe its microstructure, and subsequently the high cycle fatigue test is used to verify the surface of nanocrystallization′ s fatigue resistance.

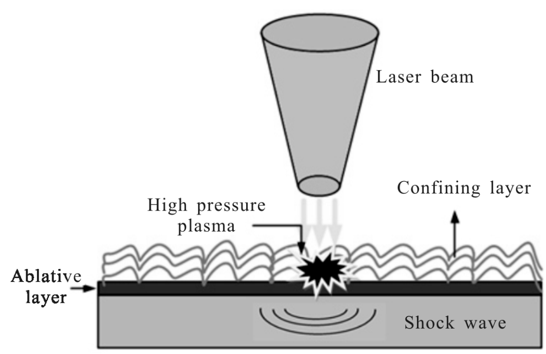


Fig.1 Schematic diagram of LSP

1 Material and experiment

The Ni-based superalloy K403 is used in this experiment. This is the material used when making a particular type of aeroengine′ s high-pressure turbine blade. Its main chemical components are shown in Tab.1.

Tab.1 Main chemical components of K403

Ni	C	Cr	Co	W	Mo	Al	Ti	Fe	B
Bal	0.11%-0.18%	10%-12%	4.5%-6.0%	4.8%-5%	3.8%-4.5%	5.3%-5.9%	2.3%-2.9%	<2.0%	0.012%-0.022%
Ce	Zr	Bi	Si	P	S	Pb	Sb	Mn	Sn
0.01%	0.03%-0.08%	<0.000 1%	<0.5%	<0.02%	<0.01%	<0.001%	<0.001%	<0.5%	<0.002%

The K403 slice specimen was machined into a size of 30 mm×30 mm×4 mm by a linear cutting machine. The specimen′s surface is progressively polished using metallographic test paper, washed with ethanol, and dried with cold wind. The center area of 20*20 is treated by LSP, shown in Fig.2.

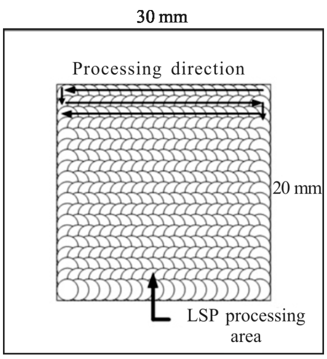


Fig.2 Processing schematic diagram of LSP

To make a contrast, we set two testing states: the original state and the strengthening state. The original state is without treatment, and the experiment for the strengthening state adopted the SGR-25-type Nd:YAG solid-state laser machine. The protective layer for laser shock is aluminum foil, with the restraint layer being water. The protective layer is used to prevent the specimen′ surface from being burned directly by laser, and the restraint layer is used to increase the shock pressure by restraining the spread of high-temperature plasma induced by the laser. The aluminum foil is replaced when the shock is repeated. The experimental parameters of LSP are as follows: pulsed laser wavelength is 1 064 nm, laser energy is 2.3-9 J, spot diameter is 1-2 mm, pulse width is 10-20 ns, shock time is 1-5 times, and spot overlapping ratio is greater than 50%. The experimental parameters of optimal LSP are shown in Tab.2.

Tab.2 Parameters of LSP

Parameters	Value
Laser wavelength/nm	1064
Pulse energy/J	8
Pulse duration/ns	20
Spot diameter/mm	2
Laser impacts	3
Lapping rate	60%
Repetition-rate	1%

The material′s microstructure was observed under the MFS-7000 X-ray diffraction instrument, X3000 scanning electron microscope, and TEM-3010 transmission electron microscope. The cross section of the sample thins up to 100 μm through mechanical lapping, and then procures a piece of Φ=3 mm as the TEM sample for observing the material′s surface organization. Notably, making the TEM sample thinner until penetration is done by single jet electropolishing, which is a solution that is composed of 300 ml CH_3OH, 175 ml C_4H_9OH, and 30 ml $HClO_4$ solution in the concentrations of 30%. For expanding the observable area, ion milling is used to do the ion thinning.

2 Results analysis

2.1 Phase composition

The phase analysis by XRD is based on polycrystalline samples′ diffraction effect through X-ray that is a method to do the determination of every components′ existing forms in specimen. The atlas analysis of XRD on the K403 specimen′s surface before or after LSP is conducted, with results shown in Fig.3.

The results of XRD analysis shows that K403′s main material is still $Ni_3(Al, Ti)$ after LSP, indicating that the LSP hasn′t induced a new phrase. The diffraction peak of specimen (111), specimen (200) and specimens′ surface after LSP is showed in Fig.3. Compared with the original specimen, the peak width at half height of Bragg diffraction significantly increases and the place of the diffraction is shifted. This works for two reasons. First, the plastic deformation induced by LSP changes the atomic distance and produces the residual compressive stress. Second, the LSP causes the segregation of the Cr element and Ti element on the surface and makes the element solid solubility change in γ' and γ causes lattice distortion, which shifts the peak position. It means the LSP makes the plastic deformation of material surface and produces the residual compressive stress that causes a mass of dislocations [8]. The paper explains the cause of the Bragg diffraction′s widening through SEM and TEM which suggests further observation on the microstructure of material′s surface.

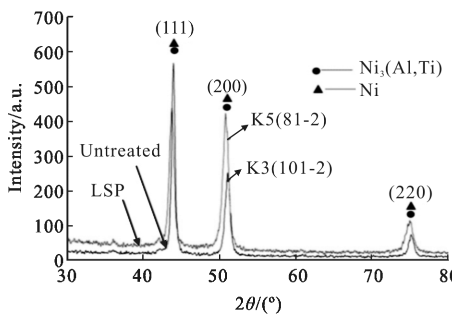
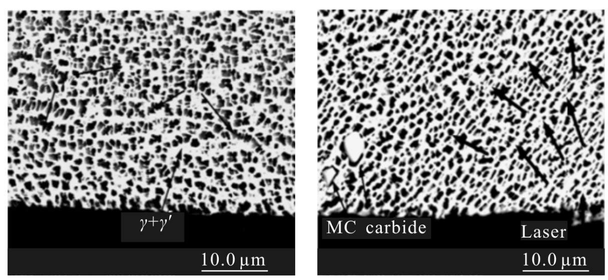


Fig.3 X-ray diffraction peak of K403 before and after LSP

2.2 Microstructure

The X3000 scanning electron microscope is adopted to observe the K403 surface organization before and after LSP. As shown in Fig.4, the casting Ni-based superalloy K403 is mainly composed of γ phase, γ' strengthening phase, $\gamma-\gamma'$ eutectic and MC carbide. The white substrate is γ solid solubility, and the black block phase, which is diffusely distributed on the γ base, is γ' phase and $\gamma-\gamma'$ eutectic. These products are produced during the alloy solidification. It is distributed on dendritic crystal, in white petal form. The block-like MC carbide is formed in a later experiment. Through comparisons between Fig.4 (a) and (b), we find that the shock wave has the reflection and transmission in the crystal surface, as the Ni-base superalloy is the biphase-polycrystal material and then it generates the multidimensional direction shock wave system. The γ phase and γ' phase are deformed to a certain extent along the shock wave system′s direction with the cell wall becoming thinner. There are two main reasons for this change. One is that only one symbol′s spare dislocation is left, as the cell wall′s different symbols′ dislocation will be cancelled out when the dislocation dimension induced by LSP comes to a certain extent. These remaining spare dislocations cause the transformation of cell structure. The second is that long stress field produced by dislocation causes the lattice distortion and show in elastic tiny distortion and atom offset, and slip dislocation causes phase boundary′s sliding and the relative displacement between γ phase and γ' phase during activity.

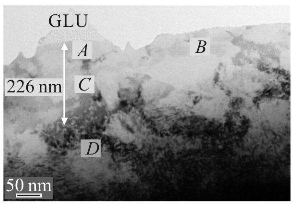


(a) Original specimen (b) Specimen after LSP

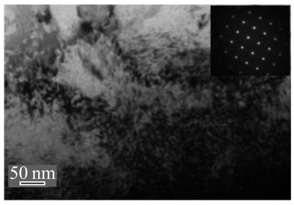
Fig.4 SEM photographs before and after LSP

Renowned from previous literature and reports, the plasma induced by LSP with high pressure(>1 GPa) and high temperature (>10^7 K) can form the high-pressure shock wave with GPa magnitude that directly acting on material surface. It makes severe plastic deformation on material surface that realizes the metal material′s surface nanocrystallization[9-10].

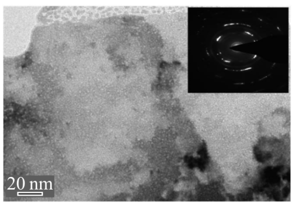
The transmission electron microscope is used to evaluate the structure and composition for observing the nanocrystalline on the surface. As shown in Fig.5, a nanocrystalline layer of 226 nanometers thick can be prepared by strengthening on the K403 specimen′s surface. Selected area *D* indicates that there is a large amount of uneven distributed dislocations formed through the plasma shock wave induced by laser shock inside the crystalline grains. Through the diffraction spots′ permutation rule known by selected area electron diffraction (SAED), we find out that the refining of the crystal does not happen here.



(a) TEM of K403 cross section



(b) Schematic diagram of selected area *D*



(c) Schematic diagram of selected area *C*

Fig.5 Transmission figure of K403

As shown in *C*′s selected area electron diffraction (SAED) pattern, the diffraction ring is continuous that shows the orientation of this layer′s nanomaterials is random. According to the scattering diffraction spots, the refining effect of the crystal on this deformed layer is more obvious.

For further observing the surface nanocrystal evolution mechanism of the K403′s surface nanocrystals, we observe the selected area *A* and *B* by high resolution, as Fig.5 demonstrated.

As shown in area *A* and *B*, there exists a typical periodic structure which is a nanocrystalline with widths measured from a few to dozens nanometers, and without a clear grain boundary. The 1-5 region (in Fig.6) have both expressed characteristics of different crystal orientation, the grain size which below 30 nm is a typical organization of nanocrystalline.

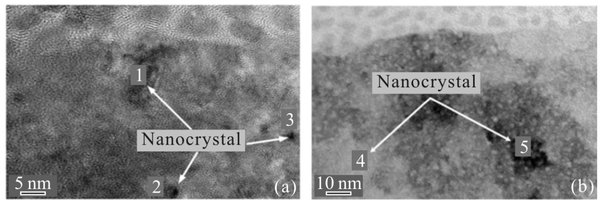


Fig.6 HTEM in area *A* and *B*

2.3 Mechanism analysis

The laser shock surface nanocrystallization is realized by laser-induced plasma shock wave. The shock wave′s propagation characteristics inside the material are related to the material dynamic response, such as pressure, wave velocity, attenuation, reflection and refraction (shown in Fig.7).

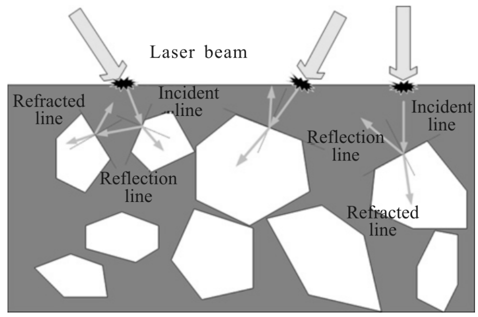


Fig.7 Schematic diagram of shock wave propagation

The high-pressure plasma shock wave is the carrier of ability to form high strain rate plastic deformation and the direct power to form the surface nanocrystallization. When the shock wave is spreading in metal material, the pressure pulse will decline, responding to the microstructure that shows the change of gradient. Meanwhile, both reflection and refraction will occur when the shock wave is in different nature interface, and the multicrystal Ni-based superalloy material exists many kinds of interfaces and defeats such as crystal boundary, phase boundary, impurities and others that induces the reflection and refraction and forms a complex wave system. When the wave pressure of the complex wave system is greater than the material′s dynamic yield strength, the increment and movement will occur within metal material. Co-currently, as shock wave′s direction is changed by reflection and refraction, the sliding in different direction increases the dislocation′s density. Thus, some interfaces inside the Ni-based superalloy easily reach the critical resolved shear stress that slip system starts to form high-density dislocation. When high-density dislocation is formed on the metal surface, the dislocation has the compatible deformation through the different movements. Additionally, the Ni-based superalloy K403, with high-level dislocation energy will easily form the cell structure by plastic deformation because the dislocation is not easy to separate in the high-level dislocation energy crystal until causing the interaction with other dislocations, where it then gathers and becomes entangled. The crystal′s dislocation after deformation is unevenly distributed; the crystal is parted into many regions of high dislocation density and low dislocation density. As the K403′s plastic deformation is mainly based on dislocation movement, it is easy to produce the dislocation cell structure. There are many dislocations in the dislocation cell, and the dislocation wall is built on the cell wall by dislocations′ interaction along with dislocations′ increment, banishment and rearrangement in a small region. The dislocation starts rotating by the lasting shock wave, and new crystal boundary is formed following the increase of the crystals′ orientation differences on both sides of the dislocation wall. The emergence of new crystal boundary is accompanied by the formation of new fine crystals, and it realizes the surface nanocrystallization, as the size of crystal reaches to the nanoscale, shown in Fig.8.

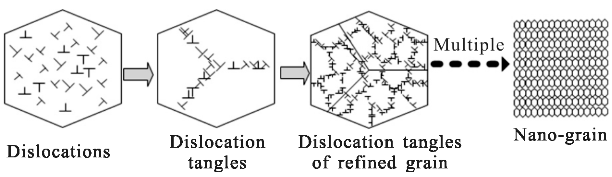


Fig.8 Schematic diagram of K403 nanocrystallization

3 Conclusion

(1) By adopting laser shock processing, a nanostructured surface layer can be prepared on the K403 specimen. The layer is about 226 nm thick and the size of the nanocrystalline becomes larger alone with the increase of surface′s depth. It will not change the phase structure by XRD, SEM and LSP.

(2) Under the laser shock processing, the surface microstructure of K403 generates a large number of dislocations and refinement grains. The closer to the surface, the small the grain is, which can be into nanoscale, ultimately showing evolution process of the dislocation slip, proliferation, devoid and rearrangement to the new grain boundary.

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