Surface & Coatings Technology 311 (2017) 337–343

Contents lists available at ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

Thermal stability of surface nanostructure produced by laser shock peening in a Ni-based superalloy

Luo Sihai, Nie Xiangfan, Zhou Liucheng, You Xi, He Weifeng *, Li Yinghong *

Science and Technology on Plasma Dynamics Laboratory, Air Force Engineering University, China

ARTICLE INFO

Article history:
Received 27 October 2016
Revised 8 January 2017
Accepted in revised form 9 January 2017
Available online 10 January 2017

Keywords:
Laser shock peening
Ni-based superalloy
Surface nanocrystalline
Thermal stability
In-situ transmission electron microscopy (TEM) annealing

ABSTRACT

A nanocrystalline layer with the grain size of about 20– 200 nm in a Ni-based superalloy was fabricated by the means of laser shock peening (LSP). The microstructure characterization of nanocrystalline layer was systematically investigated by transmission electron microscopy (TEM) and nanoindentation. And the thermal stability of surface nanocrystalline layer was studied by in-situ TEM annealing with different temperatures and duration times (500 °C/1 h, 700 °C/1 h and 900 °C/3 h). In addition, we designed a kind of nanoindentation test with the sample being post annealed at 900 °C for 10 h to verify the thermal stability of nanocrystalline layer. The results indicated that nanostructure has superior thermal stability after annealing at 700 °C for 1 h, which is higher than the dynamic crystallization temperature, 0.36 T_m (melting temperature T_m is about 1300 °C). Grain growth occurred at 900 °C, but some nano-grains with the size of around 50 nm still existed, and the nanohardness test further strengthened the evidence of good thermal stability. All these experimental results indicated that such Ni-based superalloy with nanocrystalline layer exhibited relatively good thermal stability after annealing at 900 °C. Lastly, the mechanism of thermal stability of surface nanostructure produced by LSP in a Ni-based superalloy was discussed in detail.

1. Introduction

In most cases, fatigue cracks are mainly to initiate at the surface defects of metallic components. Therefore, optimization of surface microstructures and properties can effectively improve the reliability and the service lifetime of components [1]. Due to the ultrafine grains and the large amounts of grain boundaries, nanocrystalline materials have been established to exhibit many beneficial properties relative to the coarse grain materials [2]. Many kinds of methods have been developed to obtain nanocrystalline layer in the materials surface. Such surface nanocrystallization can be realized by the means of severe plastic deformation (SPD) [3]. Laser shock peening (LSP) among SPD methods is believed to be the most common method to produce surface nanocrystalline layer without changing chemical compositions of substrate alloys [4]. Currently, LSP has already realized surface nanocrystallization for many metals and alloys, such as Ni-based superalloys, titanium alloys, magnesium alloy and stainless steel [5–11]. And the generation of high-value compressive residual stress and microstructure refinements induced by LSP are regarded as two primary mechanisms for improving fatigue properties at room temperature [5,9–12]. However, these mechanisms are only effective in service when they remain stable under the cyclic and/or thermal loading.

Ni-based superalloys, the main aeroengine turbine blade material, are always served in the presence of cyclic loading, especially thermal loading. It has an interest in extending the application of LSP on the aircraft engine parts which are exposed to high temperatures, where the temperatures can reach up to 700 °C. Z. Zhou et al. [13] investigated thermal relaxation behavior of the LSP-induced residual stress at different temperatures between 550 and 700 °C by experimental observations and finite element simulation. Y. Li et al. [5] found that 72% residual stress was relaxed after heat treatment of 900 °C/150 min. W. F. Zhou et al. [14] investigated the thermal relaxation of residual stress induced by LSP in K417 alloy at the temperatures ranging from 500 to 900 °C using experimental method. And they found that residual stress released rapidly at different temperatures in the initial 30 min, and higher temperature resulted in faster stress relaxation. I. Nikitin et al. [15] and N.F. Ren et al. [16] also had come to the same conclusion.

All in all, the relaxation of residual stress weakened the strengthening effect on fatigue performance. Hence, the thermal stability of surface nanocrystalline is the prerequisite for the LSP application on the high temperature conditions, which has attracted growing scientific interest. Huang et al. [17] investigated the microstructure evolution, phase transformation, and thermal stability of the nanograined layer of Ti-25Nb-3Mo-3Zr-2Sn titanium alloy during 300– 600 °C annealing treatment. In addition, I. Altenberger et al. [18] found that the improvement in

---

* Corresponding authors at: Science and Technology on Plasma Dynamics Laboratory, Air Force Engineering University, 1St Baling road, Xi'an, Shaanxi 710038, China.
E-mail addresses: hehe_coco@163.com (H. Weifeng), yinghong_li@126.com (L. Yinghong).

http://dx.doi.org/10.1016/j.surfcoat.2017.01.031

fatigue resistance of Ti-6Al-4 V alloy at elevated temperature are related to the high-temperature stability of highly tangled and dense dislocation substructure induced by LSP. Jia et al. [19] suggested the dislocation cells have good thermal stability in a near α titanium alloy after LSP treatment. However, to authors' knowledge, little work have been published regarding systematic research on the thermal stability of surface nanocrystalline produced by LSP for Ni-based alloys.

In this work, a nanocrystalline layer was fabricated in the surface of K417 Ni-based superalloy by the means of LSP. The microstructure of surface nanocrystalline layers were characterized by transmission electron microscopy (TEM). Moreover, in-situ TEM annealing and nanohardness tests were adopted to investigate the thermal stability of surface nanocrystalline in K417 Ni-based superalloy. Lastly, the mechanism of thermal stability of surface nanocrystalline were discussed in detail.

2. Experiments and methods

2.1. Materials

K417 Ni-based superalloy was chosen as substrate in this work, which was widely applied as turbine blades materials in aero-engine due to its excellent fatigue resistance, corrosion resistance, and creep resistance. The coarse dendritic structure with the size from several hundred micrometers to several millimeter can be found in Fig. 1(a). Fig. 1(b) shows the as cast microstructure, which mainly consists of γ solid solution, secondary γ' phase and ($\gamma + \gamma'$) eutectic. The γ solid solution is the matrix with continuously-distributed face-centered cubic structure. The large amount of γ' phase distributing among the dendrite is the main strengthening phases. The nominal chemical composition of K417 Ni-based superalloy is listed in Table 1.

2.2. Experimental process of LSP

The groove part of the turbine blade has a tendency to be broken under cycle loading in service, and it is urgently needed to be strengthened and improve its fatigue strength. Therefore, the samples of K417 Ni-based superalloy for LSP treatment were taken from the groove parts and then cut into rectangular shapes in dimensions of 25 × 20 × 4 mm (width × length × thickness), which are schematically shown in Fig. 2. LSP treatment was performed using a Q-switched self-designed Nd:YAG laser (SGR-EXTRA/25J). Prior to LSP treatment, the samples surface were polished with SiC paper with the grit number from 500 to 2400, and then ultrasound cleaning was used to degrease the surface of samples in ethanol. In the LSP process, a water layer with about 1 mm thickness was used as the transparent confining layer and an Al foil with a thickness of 100 μm was used as the absorbing layer. The detailed principle of LSP is described by Ye et al. [20]. The laser beam with a wavelength of 1064 nm and a pulse of around 20 ns was generated by laser. The paths of laser spots and LSP region are shown in Fig. 2, and the detailed laser parameters are listed in Table 2.

2.3. Heating treatment

In order to investigate the thermal stability of surface microstructure, in-situ TEM annealing at different treatment states (State I: 500 °C/1 h; State II: 700 °C/1 h; State III: 900 °C/3 h) was adopted. This was performed using a 300-kV H9500 (300 kV; point resolution: 0.18 nm), equipped with a Gatan 652 double-tilt heating holder and a Gatan 794 Slow-Scan CCD for imaging. The sample holder temperatures were calibrated in a separate vacuum system to ensure accuracy to 20 °C. The experimental parameters for the annealing processes are shown in Fig. 3.

In order to further investigate the thermal effect on mechanical properties, a heat preservation experiment was designed. Since the service temperature of K417 Ni-based alloy is over 850 °C in the aero-engine turbine blade, the samples of LSP treatment were vacuum annealed for 10 h at 900 °C with a heating rate of 20 $°C\ min^{-1}$, a pressure of 3×10^{-3} Pa and furnace cooling.

2.4. Microstructure observations and nanohardness measurement

To precisely characterize the surface microstructure of LSP-treated region, TEM observations were used. TEM was performed in JEM 2100F with the experimental parameters: FEG (field emission gun): 200 kV; point resolution: 0.23 nm and line resolution: 0.14 nm. TEM foils for the surface layers of the samples were prepared by mechanically grinding on the untreated sides to obtain thin plates with a thickness of about 20 μm; then the thin plates were twin-jet electropolished to make it suitable for TEM observations. The etchant used was 10% $HClO_4$ + 90% C_2H_5OH (vol.%).

The nanohardness measurement of the samples with different surface treatment (LSP and LSP + 900 °C/10 h) were performed by TI950 nano-mechanics test system, Hysitron Corporation USA using a Berkovich diamond indenter (maximum load is 10 mN and point resolution is 1nN, displacement resolutions are 0.04 nm and 4 nm in Z and X direction respectively). Before nanohardness measurement, the surface roughness of the cross-sectional profile must be <10 nm by mechanical grinding and polishing (surface roughness < 1 nm was obtained in this work, as shown in Fig. 4). In order to reduce the adverse effect of the nonuniform microstructure induced by LSP on the test results, multipoint indentation with the five times spacing distance of the indentation acreage was adopted along the shocked depth. In the present work, the maximum load is 10,000 μN, loading and unloading time is 5 s and the holding time is 2 s. The nanohardness results can be obtained using the model of Oliver and Pharr [21]. Three parameters were

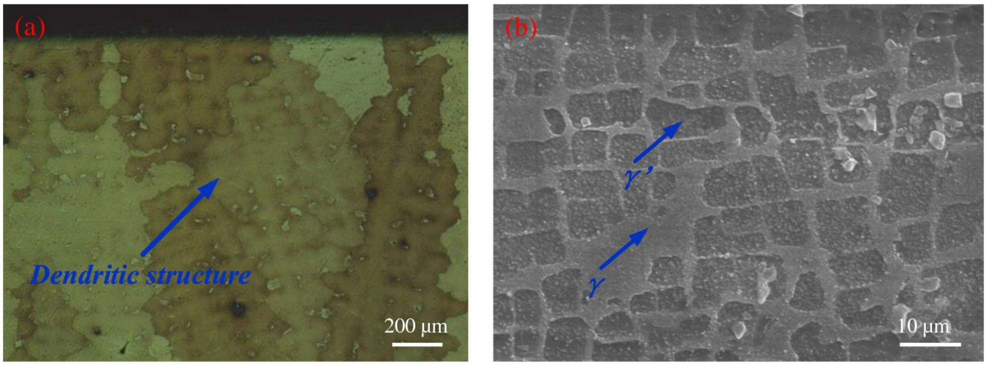

Fig. 1. The microstructure of K417 Ni-based superalloy (a) Optical microscope (OM) observation, (b) Scanning electron microscopy (SEM) observation.