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# Structure and Properties of the CrN+Cr<sub>2</sub>Ni<sub>3</sub> Layer Produced on Inconel 740 by Pulse Plasma Ion Nitriding at a Frequency of 10kHz

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# Abstract

The paper describes the microstructure and properties of the diffusive  $CrN+Cr_2Ni_3$  layer produced on the Inconel 740 superalloy by pulse plasma ion nitriding at a frequency of 10 kHz. The microstructure and the morphology of the nitrided layer were examined by scanning electron microscop, and microhardness measured by the Vickers method. It is shown that the applied layer improves properties of the Inconel 740 nickel alloy.

Keywords: Inconel 740, CrN+Cr<sub>2</sub>Ni<sub>3</sub> layer, Nitriding

# 1. Introduction

Inconel alloy 740 has good mechanical properties (tensile strength at a temperature of about 750°C -below 100MPa) and high corrosion resistance (below 2mm during 200000h), parameters which are not available with other nickel alloys such as: Inconel 617, Inconel 690, etc.<sup>1,2)</sup> These properties make it a good choice for the manufacture of tubing and piping intended for advanced ultra-supercritical pulverized coal-fired steam boilers, which are heated to a temperature of 700°C under a pressure of 375 bar.<sup>3,4)</sup> This alloy has also been investigated in terms of its application to manufacturing of the exhaust valves in automotive diesel engines<sup>5)</sup>. However, it has been suggested in ref.<sup>6)</sup> that at temperatures between 593°C and 849°C, Ni<sub>3</sub>Ti eta phase platelets form, which may adversely affect the stability of the microstructure and decrease the strength of the alloy. This drawback can be obviated by subjecting the alloys to a surface treatment. It has been found that the diffusion-type chromium nitride layer produced on the alloy surface improves its hardness, frictional wear resistance, fatigue strength, and corrosion resistance. The aim of this study was to examine in achieving the similar improvement with pulse plasma nitriding.

#### 2. Experimental Procedure

The test material was the Inconel 740 nickel alloy. It was subjected to 10 kHz pulse plasma ion nitriding conducted in a nitrogen-hydrogen atmosphere at  $560^{\circ}$ C for 8h. The surface layers obtained were investigated in an SU-70 electron scanning microscope. The phase composition was examined with a Bruker D8 X-ray diffractometer using the CuK<sub> $\alpha$ </sub> radiation. Microhardness measurements were performed by the Vickers method in a ZWICK (Materialprüfung 3212002) hardness-meter at a load of 50g.

The fatigue tests were conducted in an MTS 858 machine, model No. 359, S/N 1075319 with the +/-25kN axial force range and the +/- 200Nm torque range The machine was equipped with a TestStar II digital controller operated by the MTS TestStar v. 4.0D + TestWare-SX and 790.20 v. 4.0D and 790.20 software. The load was controlled by the force magnitude with the mean value maintained at zero and the constant stress amplitude specified for a given sample. The loading cycle was symmetric and oscillating at a frequency of 20 [Hz]. In all the samples the fatigue strength was examined at a stress amplitude of 500 [MPa].

#### 3. Results and Discussion

Figure 1 shows a cross-section of the layer produced by pulse plasma ion nitriding conducted with a frequency of 10 kHz, at temperature of 560°C for 8h. The thickness of nitrided layer can be estimated at ca.  $8\mu$ m (Fig. 1a). The layer is continuous, adheres well to the substrate with no voids at the interface. The images of the layer surface shown in Fig. 1b reveal high roughness and agglomerates of well adherent crystallites. This is confirmed by the surface roughness measurements which yielded R<sub>a</sub> exceeding 900 nm (Table 1). The X-ray phase analysis (Fig. 2) indicates that the layer contains two phases: CrN and Cr<sub>2</sub>Ni<sub>3</sub>. These phases contribute to the microhardness increase from 390 to 950 HV<sub>0.05</sub>

The Wöhler curves obtained for Inconel 740 in as-received form and after nitriding are shown in **Fig. 3**. It can be seen that, after the pulse plasma ion nitriding at a frequency of 10kHz, the fatigue parameters of the Inconel 740 alloy are evidently improved. Within the entire range of the cycle amplitudes, the failure of the samples with the  $CrN+Cr_2Ni_3$  layer occurs after a higher number of the loading cycles.

Changes in the deformation parameter  $\Phi$  at the 500 MPa load amplitude (**Fig. 4**) were used for verifying the fatigue life of the Inconel 740 nickel alloy in as-received form and after the nitriding. It can be seen that after nitriding the number of cycles to failure markedly increases which means that the fatigue resistance has been significantly improved.



Fig. 1 Cross-section, (a), and surface, (b), morphology of the  $CrN+Cr_2Ni_3$  layer produced on Inconel 740 nickel alloy.

Table 1 The sample roughness before and after nitriding.

Parameters	R <sub>a</sub> [nm]	$R_{a}$ [µm]
Inconel 740	225.15	0.29
Inconel 740 after nitriding	915.39	1.18
Notices: Rmean standard d	oviation R r	anghagag

Notices:  $K_a$  -mean standard deviation;  $K_q$  - roughness profile mean square deviation;







Fig. 3 Wöhler curves for Inconel 740 nickel alloy: (a) as-received, (b) with a  $CrN+Cr_2Ni_3$  layer.



Fig. 4 Changes of the deformation parameter versus number of cycles to failure in the Inconel 740 nickel alloy: (a) as-received, (b) with a  $CrN+Cr_2Ni_3$  layer and stress amplitude of 500 MPa.

#### 4. Conclusions

- An 8-μm thick layer composed of CrN+ Cr<sub>2</sub>Ni<sub>3</sub> can be produced on the Inconel 740 nickel superalloy by plasma ion nitriding conducted with a frequency of 10kHz and at a temperature of 560°C in a nitrogen - hydrogen atmosphere for 8h.
- (2) The layer is characterized by high hardness of ca. 950HV<sub>0.05</sub> compared to ca. 390HV<sub>0.05</sub> in the substrate material, and increased fatigue resistance compared to that of the as-received Inconel 740 nickel alloy.

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# References

- S.Zhao, X.Xie, G.D.Smith, "The oxidation behavior of the new nickel-based superalloy Inconel 740 with and without Na<sub>2</sub>SO<sub>4</sub> deposit", Surface & Coatings Technology 185, 178– 183 (2004).
- 2)S.Zhao, X.Xie, G.D.Smith, S.J.Patel, "Research and Improvement on structure stability and corrosion resistance of nickel-base superalloy INCONEL alloy 740", Materials and Design 27, 1120–1127 (2006).
- 3)N.D.Evans, P.J.Maziasz, R.W.Swindeman, and G.D.Smith, "Microstructure and Phase Stability in INCONEL Alloy 740 During Creep", Scripta Materialia, 51, 503–507 (2004).
- 4)S. Zhao, X. Xie, G.D. Smith, S.J. Patel, "Microstructural stability and mechanical properties of a new nickel-based super" Materials Science Engineering A 355, 96 (2003).
- 5)http://www.specialmetals.com/documents/Inconel%20alloy% 20740.pdf
- 6)J.P.Shingledecker and G.M.Pharr, "The Role of Eta Phase Formation on the Creep Strength and Ductility of INCONEL Alloy 740 at 1023 K (750°C)", Metallurgical and Materials Transactions. 43A, 1902-1910 (2012).