

# Effect of gas nitriding on the thermal fatigue behavior of martensitic chromium hot-work tool steel

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## ABSTRACT

The influence of heat treatment and gas nitriding on the thermal fatigue behavior of martensitic chromium hot-work tool steel was investigated. Thermal fatigue tests were carried out using a special induction heating apparatus, which consisted of induction heating and water spray cooling unit. The process of thermal cycling was simulated using a coupled heat-transfer solid-mechanics finite element model. It was seen that the thermal fatigue resistance was higher in the gas nitrided samples after austenizing at 1020 °C than for the gas nitrided samples after austenizing at 1100 °C. The thermal fatigue endurance limit was found to be maximum for the samples having a compound layer comprising of the higher phase fraction of  $\gamma'$  ( $\text{Fe}_4\text{N}_{1-x}$ ). It was also found that, lower the ratio of compound layer thickness to the total diffusion depth, the higher is the fatigue life. These results were influenced by two major effects of nitrided (diffusion) layer. First is the high compressive residual stresses imparted on the surface which tend to mitigate the effect of thermal tensile stress and secondly very high surface hardness, due to the diffusion of nitrogen, which increase the threshold for crack initiation at the surface.

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## 1. Introduction

In industrial applications, such as casting, extrusion and forging, the tooling is repeatedly subjected to cyclic thermal and mechanical loading. As a result of these severe working conditions, surface damage in the form of abrasion, erosion, corrosion and heat-checking [1] is observed. Heat-checking, which are networks of fine cracks, eventually propagate into large cracks as a result of cyclic (thermal and mechanical) loading. This leads to poor quality of the manufactured product and at times may cause premature rupture of dies. Since dies and toolings are very expensive components, their premature failure affects the bottom-line of a manufacturing industry. Several researchers have studied this phenomenon with an aim to improve the service life of dies used in forging and casting. The service life of tooling can be improved in two ways; the first method involves the improvement of the base material by adding alloying elements such as boron and niobium [2–4]; and the second method involves improvement of the surface properties to maximize the performance [5–10]. The present study is aimed at the latter approach where the complex interplay among surface hardening, residual stress and cyclic

loading parameters are studied to understand the role of surface treatment parameters on the fatigue life.

A common die material for forging and casting industries is surface hardened AISI H13, martensitic chromium hot-work tool steel. The combination of properties such as hot strength, toughness, ductility, thermal conductivity and coefficient of thermal expansion [1] provided by AISI H13 makes it a die material of choice. Since fatigue usually initiates from surface; the surface hardening treatment performed on the tooling plays an important role in determining its fatigue life. Nitriding is a widely used surface treatment process in manufacturing industries for tooling. Nitriding increases surface hardness and also induces compressive residual stresses [11–18] on the surface which are beneficial for enhancing fatigue life.

In order to estimate the effectiveness of heat treatment and gas nitriding on thermal fatigue of H13 tool steel material in accordance with actual service, it is important to establish a proper thermal fatigue testing process. In some of the previous works on experimental analysis of thermal fatigue, [6,19–24], the authors have conducted accelerated fatigue experiments for predicting and analyzing the thermal fatigue endurance limit of forging and casting dies. The parameters of thermal cyclic loading, such as temperature and cycle time for heating and cooling, used by most of these authors were assimilated in the current experimental work. In the present investigation the effect of thermal cyclic loading on crack initiation mechanisms and strain distribution

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within the material has been studied.

The effect of thermal cyclic loading on the development of strains and the eventual fatigue of the material has been studied by numerous authors. A. Fissolo et al. [23,24] studied the effect of uniaxial, bi-axial and multi-axial isothermal loading on the crack initiation mechanisms in nuclear reactor components. They proposed two models that provide the best estimate of fatigue viz., a strain-based Coffin–Manson type model and an energy-based Morrow type model. The strain-based Coffin–Manson type model has been used by many authors for predicting the maximum number of cycles for fatigue crack initiation [21,25,26]. The results of these investigations indicated that the maximum number of cycles for initiation of thermal fatigue crack under accelerated thermal loading is less than  $10^2$ – $10^4$  cycles. An FEM multiphysics model was built to study the effect of thermal loading on the stress and strain distribution in the material and to estimate their extreme values.

The aim of this study is to understand the synergetic effect of gas nitriding and thermal cyclic loading on fatigue initiation mechanisms in H13 tool steel. The effect of different heat treatments and diffusion depths of nitrided layer is also discussed in correlation with thermal fatigue initiation.

## 2. Experimental work

The material selected for present investigation is H13 hot work tool steel with the chemical composition as given in Table 1. This is martensitic steel which on tempering can attain hardness in the range of 42–46 HRC [1,27]. This hardness can be further increased by gas nitriding or any other surface hardening treatments [1,28].

The as-received material was in the form of round billet with dimensions  $\phi 90$  mm  $\times$  4000 mm. The as-received steel was cut into four pieces of equal lengths on a cutting saw and subjected to further processing. The processing sequence involved heat-treatment, machining and gas nitriding, as given below, resulting in samples with surface hardness values over 1000 HV<sub>0.1</sub>.

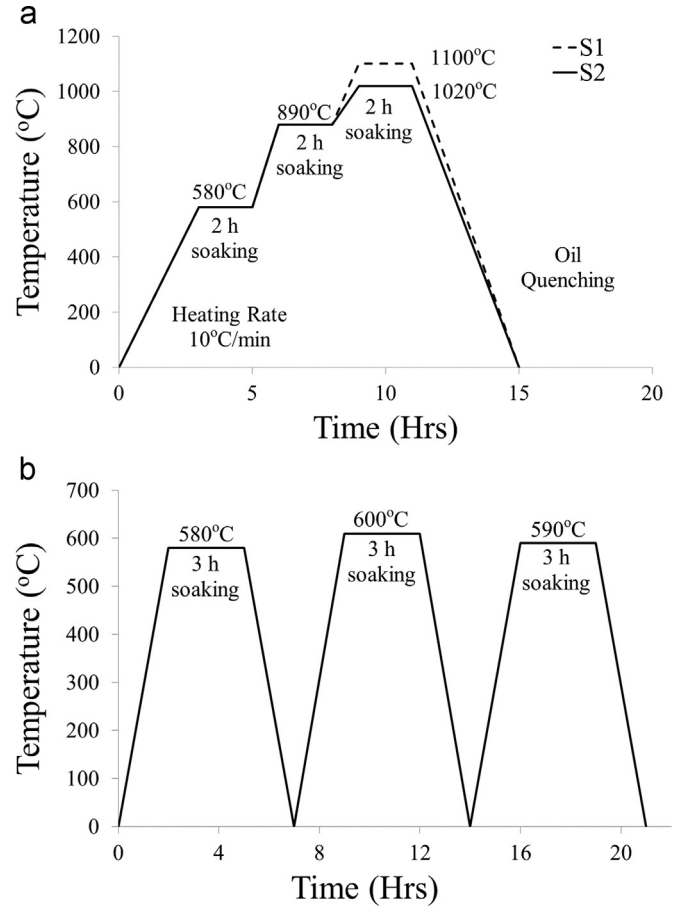
### 2.1. Heat-treatments

- Austenizing: The as received samples were divided into two batches and austenized in a closed oil fired batch furnace at different temperatures (1100 °C and 1020 °C).
- Quenching: Both the austenized batches were oil quenched to produce a martensitic structure. The detailed schedule of austenizing and quenching cycles is schematically depicted in Fig. 1a.
- Tempering: The quenched samples were subjected to triple tempering to relieve stresses and temper the martensite. The detailed schedule of tempering is schematically depicted in Fig. 1b. Hardness values in the range of 42–44 HRC were obtained in the samples after tempering.

A designation of S1 (1100 °C) and S2 (1020 °C) are used to distinguish the two batches with different austenizing temperatures (Fig. 1a).

**Table 1**  
Chemical composition of H13 hot-work tool steel.

Elements	C	Si	S	P	Mn	Cr	Mo	V	Fe
Wt%	0.39	1.15	0.01	0.01	0.26	5.22	1.16	1.11	Balance



**Fig. 1.** (a) Heat treatment cycle at temperatures of 1100 °C (S1) and 1020 °C (S2) and (b) triple tempering cycle to obtain hardness of 42–44 HRC.

### 2.2. Machining

The above heat-treated samples were turned to dimensions  $\phi 45$  mm  $\times$  60 mm with surface roughness of 0.3–0.5 Ra.

### 2.3. Surface hardening

The machined samples were gas nitriding using ammonia gas to obtain varying diffusion depths. In gas nitriding, the nitriding potential of ammonia determines the activity of nitrogen. The stronger the activity, the greater would be the diffusion depth of nascent nitrogen. Nitriding potential also has an effect on the types and the fraction of nitride phases formed during the process [16]. The nitriding potential with respect to the partial pressure of ammonia can be expressed by the formula;

$$K_N = \frac{p_{NH_3}}{p_{H_2}^{\frac{3}{2}}} \quad (1)$$

In order to reduce the amount of white layer formed by single-stage nitriding and to obtain the optimum diffusion depth (i.e. between 150 and 250  $\mu$ m as per standard industrial practice), nitriding process was carried out in two stages (also called as “Floer” process); first stage comprised low temperature and high nitrogen activity for faster initiation of diffusion; second stage comprised higher temperature and lower nitrogen activity in order to stabilize the diffusion process. These parameters also affect the formation of different iron-nitride phases. The austenized samples were subjected to three different tempering cycles. The details of the nitriding process schedule are given in Table 2. Designations of

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