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# Nitriding duration reduction without sacrificing mechanical characteristics and fatigue behavior: The beneficial effect of surface nano-crystallization by prior severe shot peening

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ABSTRACT

Generally a clear beneficial effect of nitriding duration on resultant mechanical characteristics is reported in the literature. Considering the high energy cost in the competitive business environment, this work explores any opportunities to reduce nitriding duration while not sacrificing the resultant mechanical characteristics and fatigue behavior. To this end prior shot peening is applied with particularly severe parameters to generate ultra-fine grains and nano-structures in the surface layers. It was recently shown that the local fatigue strength improvement by combination of severe shot peening and 15 h nitriding could not eventually contribute in further increasing the fatigue limit of high strength low alloy steel smooth specimens as compared to only 15 h nitriding. In the present research combination of severe shot peening with nitriding at 7.5 h is assessed. It is affirmed that improvement by hybrid treatment can be actively exploited in the form of duration reduction. The characterization is carried out by optical and scanning electron microscopy observation, micro-hardness test, surface roughness measurement and X-ray diffraction measurement of residual stress. Fatigue limit of the treated specimens is experimentally determined. A critical comparison between the hybrid process with 50% nitriding duration reduction and the original nitriding process is presented. Based on the result of this study, nitriding duration can be successfully reduced without losing improvements in mechanical characteristics and fatigue behavior if a suitable prior severe shot peening, aimed to surface nano-crystallization, is performed.

## 1. Introduction

Gas nitriding is a case hardening process whereby nitrogen is introduced into the surface of a solid ferrous alloy by holding that at a suitable temperature (below Ac1, for ferritic steel) in contact with nitrogenous gas, usually ammonia [1]. As a result released nitrogen atoms either chemically react with or diffuse between iron atoms and a case hardened surface is generated. The hardened case itself is sub-divided into compound and diffusion layers. Due to its considerable improvement in wear, corrosion and fatigue resistance, nitriding has become a well-accepted thermo-chemical process which is widely applied for high performance transmission shafts and gears, bearings, extruder screws, forging dies, injectors, crankshafts, camshafts and so on.

It has been well-known for a long time that kinetics of diffusion phenomena is highly dependent on time. Nitriding is not an exception. A clear beneficial effect of nitriding duration on resultant mechanical characteristics such as, the surface micro-hardness value [2–5], the thickness of the hardened layers [2–5], pitting corrosion resistance [2], forging die durability [6], dynamic load-ability [5], wear resistance [5] and fatigue behavior [7–9] has been reported in the literature. Therefore, prolonging nitriding may seem to be the first choice to obtain a better performance. It is accompanied, however, by the high energy cost of processing at high temperature. There is yet another alternative based on the fact that diffusion along grain boundaries is much more enhanced in comparison with the diffusion through grains. This justifies the idea of application of a prior mechanical treatment aimed to generation of defects, interfaces, increasing dislocation densities and possibly developing new micro-structure like sub-grains and eventually new grain boundaries.

For instance transformation of the coarse-grained into a very fine grained structure by high pressure torsion enhanced thickness of the nitrided layer and increased surface hardness in the subsequent radio frequency plasma nitriding of stainless [10].

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Dislocation density increment and strain induced transformation of austenite to martensite obtained by shot peening [11] were mentioned to provide faster diffusion for the subsequent plasma nitriding of stainless steel leading to twice thicker nitrided layer than not peened specimens under the same plasma nitriding condition [12]. Prior shot peening could also improve corrosion and wear resistance of gas nitrided 316L austenitic steel [13]. Generation of nano-structured surface layers through a prior surface mechanical attrition (SMAT) applied on pure iron enhanced nitrogen diffusion such that subsequent nitriding was feasible even at much depressed temperature (300 °C) [14]. Moreover, a nitrided layer twice as thick as that on a coarse-grained sample was developed on a SMAT iron sample under the same gaseous nitriding conditions [15].

Shot peening is actually a surface mechanical treatment generally applied to improve fatigue behavior of metallic parts. During the process small spherical peening media (shots) are accelerated in various kinds of peening devices to hit the surface of work piece with energy able to cause plastic deformation, compressive residual stresses and work hardening in the surface layers [16]. While a lot of attention was devoted in the last four decades to understand how residual stresses are developed during the process [17,18], it has not been a long time that shot peening was recognized as potential process to produce surface nano-crystallization. The common aspect is to use special combinations of peening parameters to multiply the kinetic energy of the shot impacts in order to generate a large number of defects, dislocations and interfaces (grain boundaries) on the surface layer of treated part and consequently transform its microstructure into ultra-fine grains or nano-structure [19]. The process in this case is called severe shot peening rather than shot peening in order to put the emphasis on the fact that it is amide to generate ultra-fine grained or nano-structured surface layers.

Based on this literature review most studies on the application of a severe plastic deformation prior to nitriding were done on stainless steel. Most recently the present authors investigated the effect of combination of severe shot peening and nitriding on the fatigue limit of a high strength low alloy (HSLA) steel [20]. The study was accomplished to shed light first on the applicability of this combination on HSLA steel and more importantly to find whether or not fatigue limit can be benefitted by this combination as much as surface hardness and case depth most often can. Based on the result, although prior severe shot peening caused up to three times deeper compound layer and produced deeper compressed layer, it was not able to further improve the fatigue limit of nitrided specimen. Local fatigue strength calculation revealed that the combination did improve the local fatigue strength up to 300 μm in depth. However, since fatigue cracks initiated below the hardened case (below 500 μm), the improvement was not seen in the final fatigue behaviour of the specimen. Therefore, the present study was designed to affirm even if the improvement did not contribute in the fatigue behaviour of smooth specimen; it exists and can be exploited in the form of nitriding duration reduction. Notwithstanding the high temperature required to perform nitriding, its duration reduction without affecting resultant mechanical characteristic and fatigue behaviour would be of great technological and scientific importance. To this end severe shot peened plus 7.5 h nitrided specimens are examined and compared with 15 h nitriding from the previous study. The treated specimens have been characterized by optical and scanning electron microscopy (OM and SEM) observation, residual stress measurement using X-ray diffraction (XRD), micro-hardness tests and surface roughness measurement. The specimens have been tested through rotating bending fatigue tests performed at room temperature. SEM observations of the fractured surfaces were applied to illustrate the failure mechanism.

## 2. Materials and methods

The material used in this study was high strength low alloy steel ESKYLOS6959 (equivalent to DIN 35NiCrMoV12-5 or AISI 4340). This class of steel is mostly used in the ground vehicle applications. Its chemical composition is summarized in Table 1. Mechanical properties evaluated through tension test are the following: 878 MPa yield stress, 1010 MPa UTS and 17.7% elongation at break. 12 Rotating bending fatigue test specimens were machined from a forging that had been quenched from 880 °C in water and then tempered at 635 °C for 5 h. The specimen geometry is presented in Fig. 1.

Specimens were subjected to severe shot peening followed by a nitriding in an industrial unit. Processing temperature and time during nitriding were 510 °C and 7.5 h respectively. Indeed the standard cycle of nitriding in the industrial unit is performed in the same temperature but for 15 h. The standard cycle of the pervious study [20] was applied. However, duration was deliberately reduced by 50% in the present study. Standard steel shots S230, using an air blast machine were employed to conduct severe shot peening. The shot peening intensity measured on "Almen A" strip was 18A. Shot peening was performed with 1000% coverage to ensure surface layers are severely deformed. The experimental assessment included OM and SEM observation of the cross section, micro-hardness measurement from surface towards depth, XRD measurement of residual stress carried out step by step by removing a very thin layer of material using an electro-polishing device, surface roughness measurement and eventually rotating bending fatigue test. The results of the in-depth residual stress measurements were corrected by using the method described in [21] to take the effect of layer removal into account. The details of the experimental procedure are excluded in this paper for the sake of brevity and readers are referred to [20] for more information. The results will be given under the label of SSP + N−7.5 h for the present samples. The corresponding results of nitriding on the same sample and in the same atmosphere and temperature but for 15 h from the previous study [20] were also added under the label of N−15 h to affirm the improvement that can be obtained by prior severe shot peening.

## 3. Results

### 3.1. Micro-structure

Overall view of the cross section by OM in Fig. 2 shows formation of a very thin compound or white layer of few microns on the top surface. The constituents of this hard and brittle layer are $\Upsilon'$ ($Fe_4N$) and $\varepsilon$ ($Fe_{2-3}N$) phases [22]. Beneath the compound layer the so-called diffusion zone with dispersed needle shape precipitates of $\Upsilon'$ in ferritic matrix as well as the solid solution of nitrogen in ferrite exists.

Formation of compound layer is more evident from the SEM image of the cross section shown in Fig. 3. Depth of compound layer was measured to be in the range of 4–6 μm after nitriding for 15 h. Performing severe shot peening prior to nitriding caused the same deep compound layer to be created even if the subsequent nitriding duration was shortened to 7.5 h. This is due to the very dense structure and fine grained surface layer generated by severe plastic deformation during severe shot peening. This can be realized by the SEM image taken from the surface of severe shot peened specimen, illustrated in Fig. 4. By severe shot peening much more defects and interfaces are generated in the surface layers through repeating impingements. With the proceeding of collisions, some areas approach to the critical condition of nano-crystallization and grain fragmentation below 100 nm occurs [19].