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INFLUENTIAL FACTORS IN THE SURFACE-HARDNESS TESTING OF A NITRIDED LAYER

VPLIVNI FAKTORJI PRI PREIZKUŠANJU TRDOTE POVRŠINE NITRIRANE PLASTI

Franjo Cajner, Darko Landek, Ivan Kumić, Amalija Vugrinčić

Faculty of Mechanical Engineering and Naval Architecture (FMENA), University of Zagreb, Ivana Lucica Street No. 1, 10000 Zagreb, Croatia
darko.landek@fsb.hr

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Nitrocarburizing is one of the frequently applied processes that significantly improve the service life of steel parts in the complex activities of mechanical loads, wear and corrosion damages. Characterization and confirmation of the quality of a nitrided/nitrocarburized layer is a prescribed norm including a determination of the surface hardness, nitriding hardening depth, compound-zone thickness and its porosity. In the testing of the surface hardness, in spite of determined conditions, there are additional factors that can affect the obtained result and can lead to a misunderstanding between a customer and a provider of the service of nitriding. In this paper, possible influential factors related to the surface hardness are considered and statistically analyzed by the ANOVA two-factor test. The considered influential factors are: the surface-preparation method and the loading force. The tests carried out during the research indicated a significant influence of the loading force and of the surface preparation on the results of the nitrided/nitrocarburized-steel surface hardness. Also, the indentation size effect is confirmed in the hardness tests with a small loading force.

Keywords: nitriding/nitrocarburizing, surface hardness

Nitrocementacija je pogosto uporabljen postopek, ki občutno izboljša zdržljivost jeklenih komponent pri kompleksnem delovanju mehanskih obremenitev, obrabe in korozijskih poškodb. Karakterizacija in potrditev kvalitete nitrirane/nitrocementirane plasti je predpisana in vključuje določanje trdote površine, globino utrjevanja z nitridi, debelino spojinske cone in njeno poroznost. Pri preizkušanju trdote površine so kljub točno določenim pogojem prisotni še dodatni faktorji, ki lahko vplivajo na rezultate in lahko povzročijo nesoglasje med uporabnikom in izvajalcem nitriranja. V tem članku so mogoči vplivni faktorji obravnavani in statistično analizirani z ANOVA-preizkusom dveh faktorjev. Obravnavana faktorja sta: metoda priprave površine in sila obremenjevanja. Preizkusi so pokazali močan vpliv sile obremenjevanja in priprave površine na rezultate trdote nitrirane/nitrocementirane površine. Pri merjenju trdote z majhnimi obremenitvami pa je bil ugotovljen tudi vpliv velikosti odtisa.

Ključne besede: nitriranje/nitrocementiranje, trdota površine

1 INTRODUCTION

Nitrocarburizing is a thermochemical process for modifying the work-piece surface where carbon and nitrogen are diffused into the surface to form a surface layer consisting of a compound layer and a diffusion layer.[1] The ISO 15787:2001(E) standard prescribes drawings and characterization of the nitrocarburized layer. The characterization of the layer includes the surface-hardness testing, determination of the effective depth of nitriding, thickness of the compound layer and its porosity.[2] The above mentioned standard is supplemented with the ISO 6507-1:2005 standard, according to which the hardness should be tested with the Vickers hardness test method.[3] However, even in the case when all the requirements of these standards are fully satisfied, the results of a nitrocarburized-layer characterization may differ due to a number of influential factors. These differences in the results may lead to a misunderstanding between a customer and a provider of the service of nitriding. Here, the inaccuracies and imprecision of a surface-hardness testing are of special importance. The results of a nitrocarburized-layer surface hardness testing are significantly affected by the following factors: indentation load of the indenter, accuracy and precision of the hardness tester, preparation of the test-sample surface, porosity of the compound layer, and the measurer's experience.[4]

2 EXPERIMENTS

In order to determine the effect of surface preparation and of the applied load on the results, hardness tests were carried out on the surface of the normalized annealed 21CrMo5-7 steel after it had been nitrocarburized in a TENIFER salt bath (580 °C/2 h oil cooling). Before the hardness tests, the surfaces of the nitrocarburized samples were prepared by grinding and polishing according to **Table 1** (the treatments marked as: A, B, C). The aim of the polishing was to reduce or remove the porous part of the compound layer in order to reduce the adverse effect of the porosity on the hardness test results. The hardness of the nitrocarburized layer was tested with the Vickers hardness test method under three different loads: 4.9 N, 9.81 N, and 49.03 N (HV0.5, HV1, and HV5). The nitriding hardness depth (NHD) was determined on a cross-section of a metallographic test sample with the Vickers test under the load of 4.9 N (HV0.5) according to the ISO 15787:2001(E) standard. Optical microscopy was applied to the same sample to determine the thickness of the compound layer and its porosity.

Table 1: Preparation of the test sample surface for hardness testing
Tabela 1: Priprava preizkusne površine vzorca za merjenje trdote

Sample	Sandpaper grain size/duration (min)
A	P1000/10
B	P1000/10 + P2000/5
C	P600/5 + P1000/5 + P2000/5

3 RESULTS

The surface-hardness test results for the nitrocarburized samples prepared with three different surface treatments and with three different loads applied are presented in **Figure 1**. The nitriding hardness depth (**Figure 2**) is 0.22 mm. From the same figure one can see that the hardness decreases sharply with an increased distance from a sample edge. An analysis of variance (ANOVA) was carried out to determine the significance of the surface preparation and scale loading on the results of the hardness tests conducted on a sample surface. The ANOVA results (**Figure 3**) confirmed the significant influence of the surface pre-treatment and the selection of the indentation load of the indenter on the results of hardness tests.

Generally, lower hardness values were measured when a larger amount of the material was removed from the surface of a nitrocarburized sample, i.e., when sand paper with larger grains was used before the testing. The nitrocarburized layer is thin and the hardness decreases sharply with an increased distance from a sample edge.

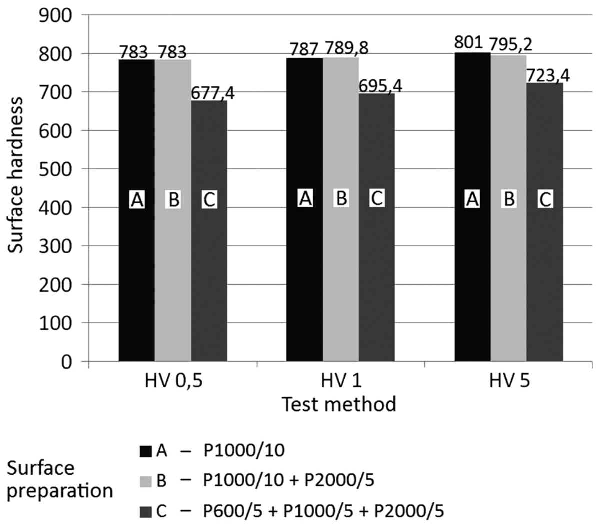

Figure 1: Surface hardness of nitrocarburized 21CrMo5-7 steel samples after different surface preparations (**Table 1**) and applied loads
Slika 1: Trdota površine nitrocementiranega vzorca iz jekla 21CrMo5-7 pri različnih pripravah površine (**Tabela 1**) in uporabljenih obremenitvah

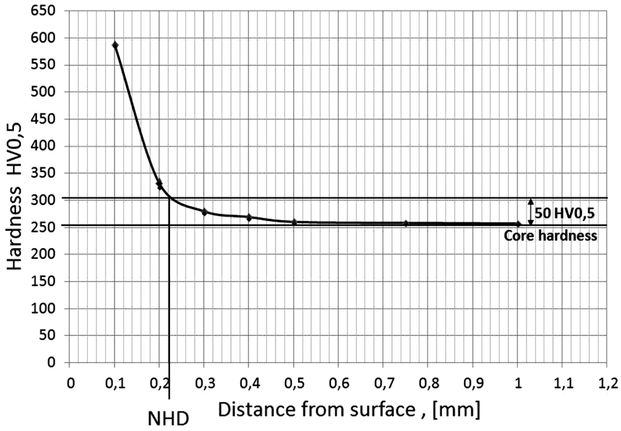

Figure 2: Hardness distribution on the cross-section of a nitrocarburized EN 21CrMo5-7 steel sample
Slika 2: Potek trdote preko prečnega prereza nitrocementiranega vzorca jekla EN 21CrMo5-7

Therefore, if the hardest part of the layer is removed by grinding, the hardness of the surface is thus reduced. Although samples A and B have been treated in a similar way, sample B, which has been additionally treated with

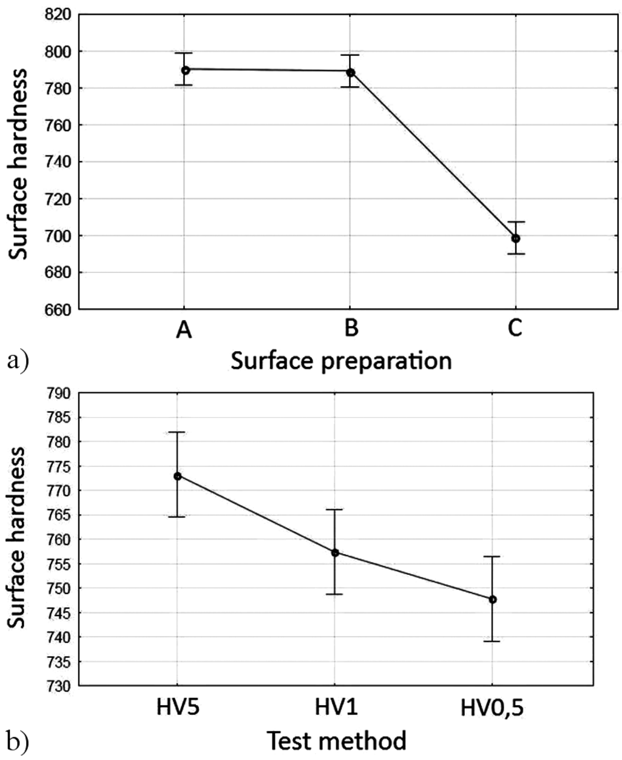

Figure 3: Influence of the surface preparation and test load on the measured values of the surface hardness on the same nitrocarburized EN 21CrMo5-7 steel sample: a) effect of the surface condition on the measured values of surface hardness, b) effect of the indentation load of the indenter on the measured values of surface hardness
Slika 3: Vpliv priprave površine in obremenitve pri preizkusu na izmerjeno vrednost trdote površine na enakem nitrocementiranem vzorcu jekla EN 21CrMo5-7: a) vpliv priprave površine na izmerjeno vrednost trdote površine, b) vpliv obremenitve pri vtiskovanju vtisnega telesa na izmerjeno vrednost trdote površine

finer grade sandpaper, exhibited a better reflection of the light, which made the measurement of diagonals easier, while the hardness of the surface remained almost the same. Accordingly, the surface preparation of sample B could be considered optimal (as it shows a good reflection of the light along with the preserved good properties of the nitrocarburized layer).

If we consider the change in the surface hardness in dependence on the indentation load of the indenter, we can notice that higher values of hardness are obtained when higher loads are applied. This is in line with the expectations that the hardness measured under low loads will depend greatly on the applied force due to the short diagonals of indentations, which can hardly be measured accurately. This phenomenon, known as the *Indentation Size Effect* (ISE), is of significance at low loads.[4] The compound-layer thickness (CLT) of sample A was approximately 16 μm, with a porous part of approximately 8 μm. The mean CST of sample B was approximately 15 μm and it was reduced to only 5 μm on the damaged spots produced by rough grinding. If the ground surfaces of samples A, B, and C are compared using a light microscope, one can notice that a part of a compound layer has been removed in the process of surface preparation. The rougher surface of sample C after the surface preparation (the surface is damaged, more material has been removed) is the most probable cause for the values of the surface hardness that are lower than those of sample B.

4 CONCLUSION

The following conclusions can be drawn from the investigation into the effect of a surface treatment and indentation load of the indenter on the test results for the nitrocarburized EN 21CrMo5-7 steel surface hardness:

- A nitrided layer with the compound-layer thickness (CLT) of 16 μm and with the porosity of 8 μm was obtained. The nitrided hardness depth (NHD) was 0.22 mm.
- The values of the surface hardness (according to the Vickers test) greatly depend on the quality of the surface and the applied load. By using sandpaper with larger grains to grind the spot to be measured, the porous part of the compound layer is removed, the layer becomes thinner and the measured values of the surface hardness are lower. When the loads of 0.981 N and 4.89 N were applied, higher values of the surface hardness were obtained for the same measurement spot with the load of 0.981 N than those obtained in the test with the load of 0.489 N.
- During the preparation of the hardness testing of the compound-layer porous part, a compromise between grinding and polishing has to be made in order to obtain a good light reflection and to preserve a sufficient thickness of the surface layer.

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