EFFECT OF NITRIDATION ON CONTACT FATIGUE AND WEAR DAMAGE OF ASTALOY CrL AND CrM STEELS

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Abstract
Contact fatigue of sintered Astaloy CrL and CrM type steels with 0.3-0.7%C additive after nitridation in gas was investigated using pin-on-disk technique and related to results obtained by wear testing. The results of contact fatigue resistance were evaluated in terms of Wöhler's curves at 50.10^7 cycles. Fatigue limits determined by this way, which were between 1080 and 1575 MPa, follow the hardness HV10 of the nitrided surface. Such surface is covered by so-called ε-phase. Its hardness increases with increasing carbon content as well as with Cr/Mo content in the material. Influence on the contact fatigue lifetime is relatively small which can be attributed to other negative properties of this hard layer. This is supported by metallographic microscopic analysis and also by character of its brittle fracture damage. The main difference in damaging of materials produced in PM way, in comparison with classical wroughts, is the quantity of pittings. In the compact materials there is largely one. In PM materials little infringements occur practically all around materials track circumference.
Wear testing did not yield unambiguous results. They were not in direct accordance with contact fatigue results. The two damage mechanisms are based on different principles. Behaviour of PM materials is not sufficiently explored at present. It appears that the wear is more related to microhardness / type of resulting carbides and their exclusion, evenly in the grain or grain boundaries /. It is necessary to verify this supplementary observation by targeted laboratory procedures.

Keywords: Cr- sintered steels, Contact fatigue, Wear, Surface modification

1 Introduction

Recently, the development of PM materials advanced to the point where replacement of compact materials by sintered ones is possible. This replacement is feasible without implementation of special treatment techniques. The materials can be used immediately after sintering either in case of classical cooling or in case of rapid cooling (sinterhardening). However, there are cases when basic mechanical properties do not suffice and additional treatment is necessary, above all to improve surface resistance. Especially, there are cases of specific applications, where high contact fatigue and wear (abrasion) resistance are required. In such cases usually the influence of well proven methods of surface strengthening, such as case-hardening and nitridation, are explored. Actual situation is promising, because new materials, in which properties close to that of classical compacts have been achieved, are being introduced to engineering practice. At present among such materials, commercially available, belongs to prealloyed Astaloy powders

CrL and CrM which, after appropriate preparation and processing, exhibit properties useful in manufacturing of highly stressed machine components, for instance gears. Currently, research of contact fatigue in this type of material is underway [1-5]. Similar reasoning applies to research on wear. [6-9].

The aim of the present work is to assess the resistance of these two types of materials with two different levels of carbon after nitridation against contact fatigue and wear so that not only strictly engineering parameters are determined but, based on metallographic–microscopical analysis, to understand damage mechanisms and other phenomena related to this type of exposure. The obtained results will be used for comparisons with other treatments of these material systems, which is the subject of the project of the Ministry of Education and SAS Vega 1/0464/08 "Tribological aspects of failure of sintered materials with an emphasis on contact fatigue and wear". The treatments, which will yield the best results and which will likely further improve resistance to contact fatigue and wear will be subject to further research leading to technologies usable in manufacturing processes.

2 Experimental materials and procedure

The experimental materials were ferrous powders Astaloy CrL (Fe-1.5 Cr-0.2 Mo) and CrM (Fe-3Cr-0,5 Mo) types, from which after adding 0.3% C or 0.7 % C as well as 0.5% lubricant of HW type, the samples with dimensions ϕ 30 x 5 mm were die pressed. The pressure was 600 MPa. The samples were sintered in an atmosphere composed of 90% N_2 + 10% H_2 at 1120°C / 60 min. Against possible oxidation the atmosphere had been frozen down (dew point -57 °C) and samples were stored in a retort within powder bed of Al_2O_3 + 5% C. In this way prepared samples were machined to ϕ 28 mm with central circular opening ϕ 10 mm and then subjected to heat treatment which consisted of austenitization (Mat.1,3) at 860 °C/15 min and (Mat. 2,4) at 800 °C/15min.) followed by cooling outside the furnace space. After this heat treatment, the samples were nitrided in: 560 °C / 160 min. - ammonia, + 20 min. in nitrogen. The content of ammonia removed from the furnace was 40%. The samples were subjected to standard tests of hardness, metallographic analysis, and contact fatigue tests. The fatigue testing was carried out on equipment AXMAT - see **Fig.1a**.

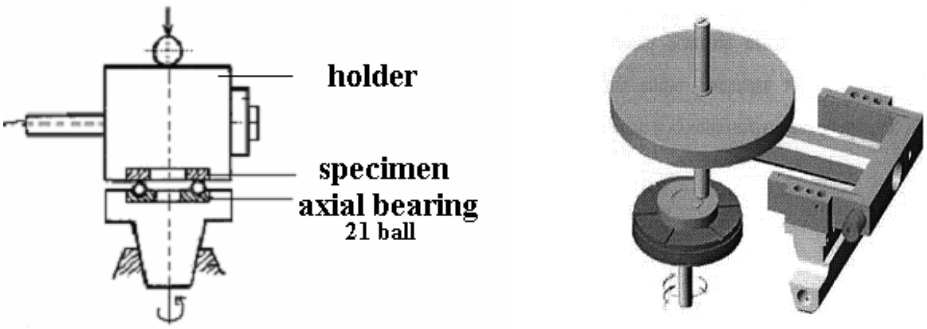

Fig.1 a,b Principle of contact fatigue equipment – AXMAT and CSM

Equation $\sigma_{\max} = 0{,}388.\sqrt[3]{4.F.\frac{E_1^2.E_2^2}{(E_1+E_2)^2}.\frac{1}{R^2}}$, according to [10], was used to calculate the Hertz stress values, where F is the applied force, E_1, E_2 are the Young modules of materials used and R

is the radius of the rolled ball. The frequency was about 500 cycles / sec. As lubricant gear oil SAE 80th Mogul was used. The results of contact fatigue are presented by classical S-N curve, where lifetime was related to the value of 50.10^6 cycles. The wear tests were performed on the CSM tribometer, **Fig.1b**, under the following conditions: 8 N load, speed 20 cm/s, distance of 360 m, in dry sliding and in the same lubricant. Indenter (pin) was a steel ball with 5 mm diameter.

3 Results

Four experimental materials were studied. Their chemical composition and some mechanical properties are in **Table 1**.

Table 1 Experimental materials and their properties after nitridation

Mat.	Chemical composition	γ (g.cm^{-3})	HVm 0,05	σ_C(MPa)	HV10	HV10/σ_C
1.	1,5Cr+0,2Mo+0,3C	~ 7	792	1080	376	0,34
2.	1,5Cr+0,2Mo+0.7C	~ 7	839	1330	400	0,30
3.	3Cr+0,5Mo+0.3C	~ 7	966	1400	450	0,32
4.	3Cr+0,5Mo+0.7C	**~ 7**	1036	1575	512	0,32

Fig. 2 shows the results of the contact fatigue testing expressed as Hertzian stress vs. number of cycles. The fatigue limit values at 5.10^7cycles are compared and are given in Table 1. The plot shows that the fatigue limit increases with increasing carbon and chromium contents. It is in agreement with expected influence of chemical composition on mechanical properties (in our case it is also documented by hardness values – see Table 1, macrohardness HV 10 was measured at the surface and microhardness HV 0.05 in the cut perpendicular to the surface - Fig.5. This trend can be well described by a ratio HV10/σ_C which is 0.32 ± 0.02.

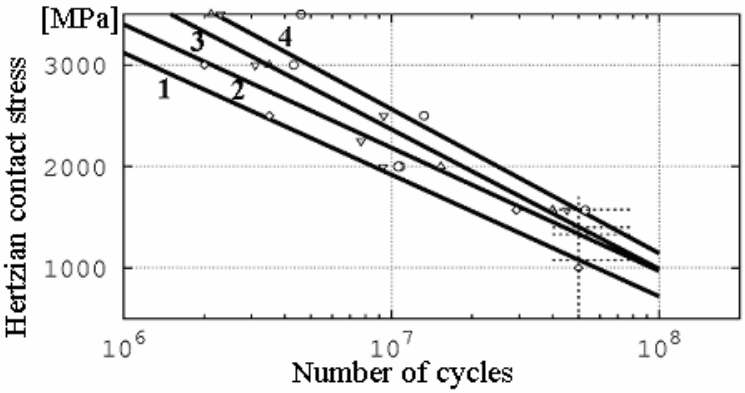

Fig.2 Pitting as function of number of cycles on applied stress

The surface destruction in contact fatigue is determined by pitting. Basically it is pull-out of material from the surface. In case of compact materials usually only one pitting appears. In PM materials several pitting sites appear along the running track. It is related mainly to the way of their identification, size and mechanism of creation. In our case the pitting sites differ neither qualitatively nor quantitatively. Their typical appearance and character is illustrated in material CrM+0.7C – **Fig.3**. In the photograph also the width of the track made by the rolling balls is visible. Metallographic observation showed that pitting forms gradually. Applied stresses cause

microfracturing of the surface nitride layer which leads to formation of surface cracks – shown by arrows in **Fig.4**. After the material had been pulled-out, the surface of the pitting has morphology as it is shown in the right-hand side of the picture.

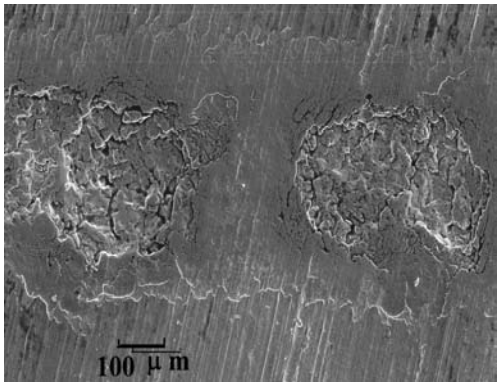

Fig.3 Morphology of pitting

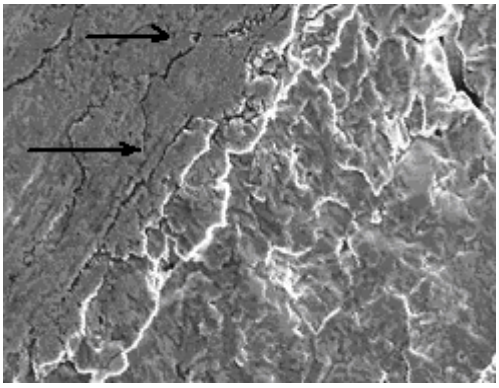

Fig.4 Transition between surface with cracks and pitting

Surface layer in common nitrided materials are in general well explored, though not all nuances may be known [10]. This layer consists of a sequence of several phases. In PM materials nitridation has not been used widely yet. Because of their porous microstructure its nature is rather complicated and has not yet been satisfactorily described, it can be even said that for CrM and CrL such description is absent. For engineering purposes the nitridation is evaluated basically only by the hardness and thickness of the nitride layer. The results of measurement of these variables for our materials are depicted in **Fig.5**. The maximum hardness was from 792 to 1036 HVM 0.05 for depths about 100-150 μm. The ε phase, formed on the surface of compact materials, is hexagonal nitride with variable composition $Fe_{2\text{-}3}N$. In alloyed, isothermally saturated steels two distinct layer were identified – a thin surface layer $(Fe,M)_2(N,C)$ and a subsurface one $(Fe,M)_3(N,C)$.

As shown by microscopic analysis, on the surface of all our materials the mentioned ε phase, 15 to 20 μm thick, had been formed, Fig.6. Its hardness was slightly lower, 500 – 960 HVM 0.05. Detail analysis of the ε phase showed also its structure and the mechanism of its fracture failure – **Fig.6a,b**.

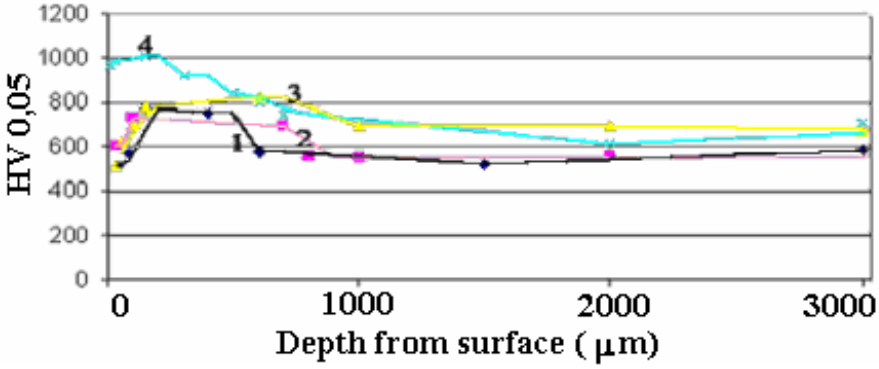

Fig.5 Depth profile of microhardness measured on cross sections of the nitrided materials

During contact fatigue exposure of the nitrided PM materials, fracturing of their hard surface layers occurs, particularly in places with a pore or a brittle particle, or close to them. Then delamination of the ε layer takes place – **Fig.7**. Simultaneously, the same stresses lead to formation of new cracks in certain depth under the surface, in accordance with theory.

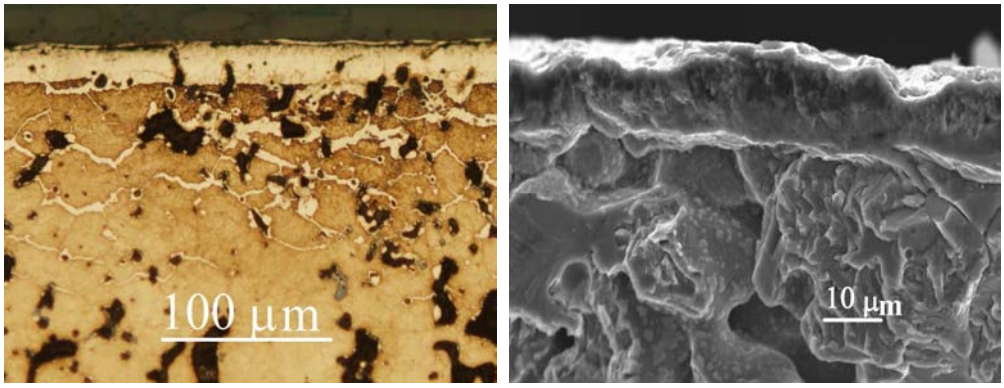

Fig.6 a,b Nitrided layer on the cross sections of the materials 4 and 1

Both mechanisms are well visible in **Fig.8**. Failure, i.e. form ation of pitting occurs already at relatively low stresses, mainly due to the fact that the main mechanism of cracking of the nitrided layer is brittle fracture – **Fig.9**. This can take place between two pores or again in places where hard particles are located.

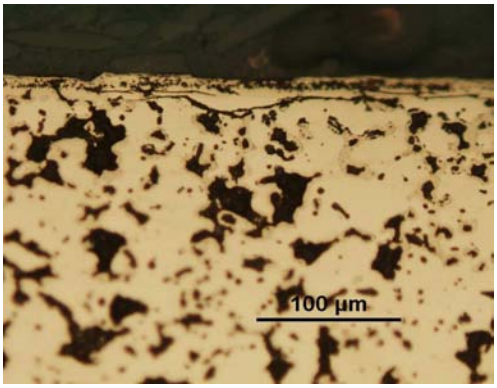

Fig.7 Cracking and delamination of the ε layer

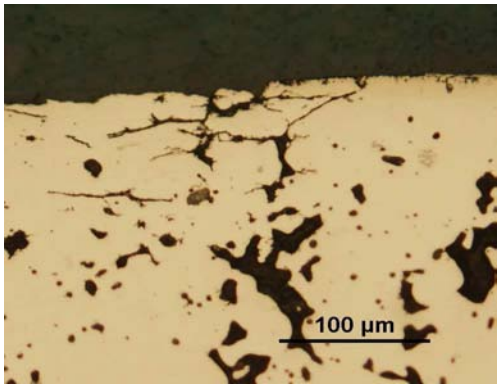

Fig.8 Crack formation under the surface

Wear testing by means of the pin-on-disc technique was performed using the same lubricant as the contact fatigue experiments. In **Fig. 10** the dependence of the overall wear (integral wear depth) on sliding distance is shown. The measured wear is a sum of the worn cap of the indenter and of the wear track in the surface of the sample. The height of the worn cap is calculated from the size of worn circle visible on the indenter surface. Subtracting this value from the measured (integral) depth the wear of the sample is calculated. This result is in **Fig.13**. In **Fig.12** friction coefficient along the sliding distance is illustrated. The lowest values of the friction and wear were found for the material CrM+0.3C.

Decrease of the friction coefficient can be explained with the help of **Fig.11**, where it is clear that predominantly it was the indenter where the wearing took place. This is caused by the fact,

that the hardness of the nitrided layer (here mircohardness has to be considered) is higher than that of the indenter. Because of that the contact area increased and, while the load was constant, the actual pressure decreased. These conditions then probably enabled formation of continuous oil film between indenter and sample. This is in agreement with micrographs of the wear damage of indenter and samples. The worn surface of the indenter is a circle with approximately 600 μm diameter – Fig.13a, which corresponds to height of about 19 μm. This would mean the depth of the wear track in the sample of about a tenth of a micrometer – Fig.13b. This is practically immeasurable and under microscope nearly unidentifiable.

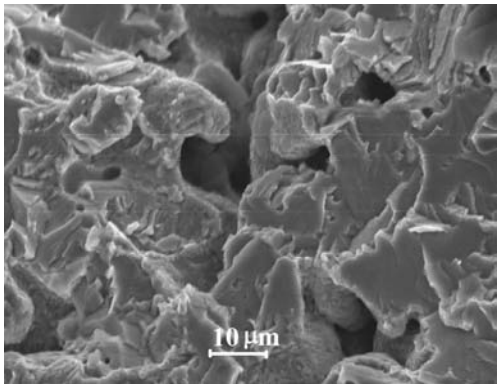

Fig.9 Brittle fracture of nitrided layer

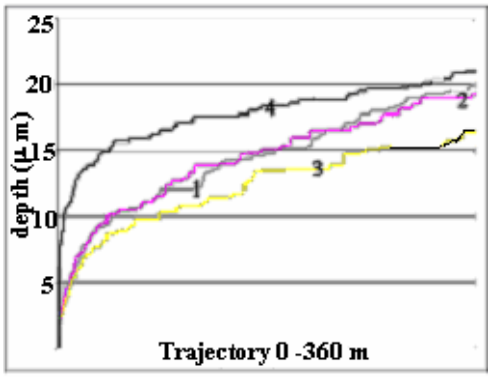

Fig.10 Wear of indenter and sample expressed as depth in micrometers

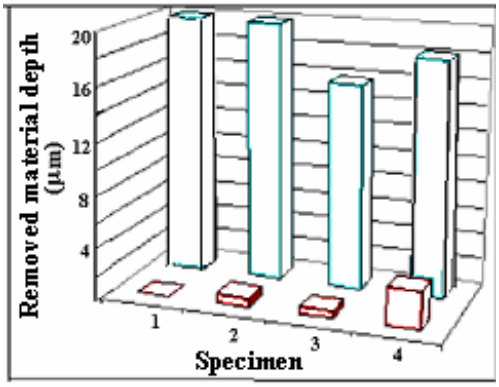

Fig.11 Comparison of wear of sample and indenter

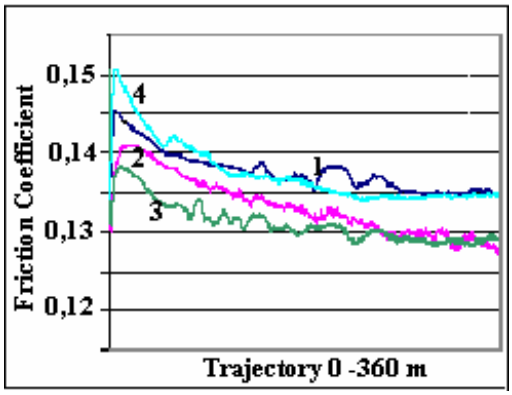

Fig.12 Coefficient of friction as function of sliding distance

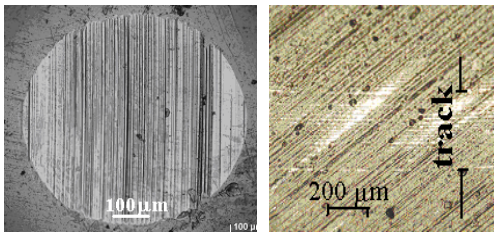

Fig.13 a,b Micrographs of worn areas in indenter and sample 1

4 Discussion

The surface strengthening of the four material variants by nitridation has lead to expected results. Hardness and thickness of the nitrided layer was sufficient to assume that this surface treatment would bring significant improvement of contact fatigue resistance. Thank to the effect of hardness of surface nitride layer it increased of about 30 %, when compared to CrL+0.3C or CrM+0.7C. However, it seems that the absolute values achieved by nitridation, when compared to as-sintered or case-hardened systems (the study of sintered materials will be published shortly, that of the case-hardened ones are in [12]), improved only marginally, despite the 100% increase of surface hardness by nitridation (from 168-253 up to 376-512 HV10). The reasons are to be found in specific properties of the nitrided layer. The ε layer formed on surface has lower hardness than layers (phases) that follow it, α,γ ... etc., that's why in practice it is usually removed by grinding or, before finishing the nitridation, by removal of nitrogen from the ε phase to α by keeping the material in N_2 atmosphere. This was done in our case. However, metallographic analysis suggests that 20 minutes was not sufficient. Preliminary tests, after we had ground this layer away, showed no improvement.

In literature we have not found description of effect of classical nitridation on the contact fatigue. In [13] a study of PM materials is described but it concerns carbonitridation or plasma nitridation, respectively. Similarly, the results are not very compelling. It will be necessary to do further careful studies in order to find real causes of small effect of classically nitrided layer on the contact fatigue properties of PM materials which leads to their low fatigue resistance.

5 Conclusions

1. It has bee confirmed that the contact fatigue properties of the experimental PM materials are function of hardness of the surface layers. Its actual values however depend on the microstruc-ture after nitridation and related brittle fracture character. Fracture shows clear signs of brittle cleavage facets.
2. Cracks leading to pitting originate on the surface as well as under it, in sites with maximum Hertzian shear stress.
3. The nitridation improves the wear resistance of the experimental materials, the differences between the material variants were insignificant.
4. It seems that wear does not significantly affect the contact fatigue properties.

Acknowledgements

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