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Plasma nitriding and its influence on contact fatigue of sintered steel

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Abstract

Plasma nitriding treatment in combination with shot peening is a promising way of material processing to improve contact fatigue resistance of PM components made of prealloyed Astaloy CrL powder. This powder was used to manufacture four material variants differing by carbon contents 0.3 % and 0.7 % C, and by surface status (as received and shot peened). Tests were done on a pin-on-disc type device and evaluated in terms of formation of pittings at 50×10^6 cycles. They revealed the positive effects of plasma nitriding. In the material with 0.3 % C, the fatigue resistance increased by 65 % and if the nitriding was preceded by shot peening, it increased by 172 %. In the material with 0.7 % C, the corresponding increase was 38 % and 107 %, respectively. The surface nitrided layer, the mechanisms of its damage, and causes of pitting formation and growth depending on Hertzian stresses are described.

Key words: Cr-sintered steels, contact fatigue, surface modification, pittings

1. Introduction

Using products and engine parts manufactured by PM is becoming a generally accepted method of replacing the more expensive and complex versions of products made of compact materials. It is also supported by the commercial success of a material basis which meets the required parameters. First of all, it includes the material manufactured by the Höganäs Company under the brand names of CrL or CrM Astaloy. These pressure casting powders are alloyed by chromium and molybdenum together with carbon and offer very good mechanical properties making them interchangeable with compact steels.

In terms of their properties, much effort was devoted to special case applications, such as engine parts and spare parts of roller bearing types or gear wheels. Conducting research in this field requires knowledge of the contact fatigue resistance of the materials in question. Nowadays, the issue is enjoying worldwide interest on the part of material research [1–5]. Generally, there are known some ways of how to improve resistance to contact fatigue, and currently the focus is on specific technologies, particularly on finding the quantitative differences. Of course, scientific and technical justification of these results is indispensable. Surface hardening is one of the methods suitable for increasing resistance against contact fatigue [6–10]. This article is aimed at investigation of the effect of plasma nitriding and surface shot peening on the contact fatigue resistance of the Astaloy CrL material.

2. Experimental materials and methods

The samples were prepared using the ferrous, commercially produced powder CrL (Fe-1.5%Cr-0.2%Mo) of Astaloy type. Chemical analysis of this powder showed the following composition in wt.%: 1.37 Cr, 0.2 Mo, 0.012 C, 0.156 O_2, 0.0025 Al, 0.02 Si, 0.003 S. Mass losses after sintering were 0.16 % and the rest insoluble in HCl 0.206 %. By adding 0.3 % and 0.7 % of carbon, two sets of samples were produced. After admixing the HWC type lubricant, the resulting powder mixtures were compressed by 600 MPa and samples with dimensions ø $30 \times 5\ \mathrm{mm}^2$ were prepared. These were subsequently subjected to sintering in an atmosphere composed of 90 % N_2 + 10 % H_2. The sintering

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took place at a temperature of 1120 ℃ lasting for 60 min. To prevent potential oxidation of samples, the atmosphere was frozen (dew point –57 ℃). The samples were placed in a retort with a charge of a mixture of Al_2O_3 + 1.5 % C for samples with 0.3 % C contents and 3 % C for samples with 0.7 % C as a protection against possible decarburization. Sintering was followed by natural cooling of the samples in the retort with protective atmosphere outside the furnace. The samples produced were then mechanically machined to the outer dimension of ø 28 mm with an internal hole of ø 10 mm. The samples prepared in this way were then subjected to grinding from both sides to ensure their regular shape and to prevent undesired vibrations during testing. Finally, half of the samples were subjected to shot peening by ferrous granulate of ø 0.6 mm. The spraying device operated at a rate of 7000 rpm, shot peening took place at an angle of 90° with respect to the sample surface. On both sides of the sample, 9 kg of granulates were used. Both types of samples, shot peened ones and those without shot peening, were then subjected to two-sided plasma nitriding (at the University of Defence, Brno) under the following conditions: temperature of nitriding 500 ℃, proportion of the hydrogen: nitrogen gases 24 : 8, pressure 2.8 mbar, voltage 520 V, pulse length and pulse interval 100 µs, time of nitridation 35 h. The four sets of samples having undergone the preparation as described above, were subsequently subjected to contact fatigue tests (using the pin-on-disc system) using the AXMAT type device – Fig. 1, operating at 500 rpm. The sample replaces the upper part of the axial bearing, with 18 balls of ø 3.969 mm size, made of bearing steel, rolling on its surface. The balls are located in the lower part of the axial bearing and are lubricated with MOGUL SAE 80 transmission oil, circulated and constantly filtered during testing. Apart from that, measurements were also extended to the hardness of the HV10 and HRB on their surfaces as well as the microhardness on the cross-sections to determine the effect of shot peening. Metallographic microscopic analysis was carried out using scanning electron microscope (SEM) with attached EDAX analyzer.

For data integrity and accuracy of the calculation of Hertz stress it is to be noted that in the formulas for calculating them in the case of materials with $\gamma = 7\,\mathrm{g\,cm^{-3}}$, Young's modulus of 140 GPa was used. For the shot peened samples, where the surface layer has a higher density 7.4–7.5 $\mathrm{g\,cm^{-3}}$, the value of 180 GPa was used, according to the equation:

$$E_2 = E_1 \left(\frac{\gamma_2}{\gamma_1}\right)^{3.4} \qquad [11].$$

For the compact material of steel balls, this value is 210 GPa.

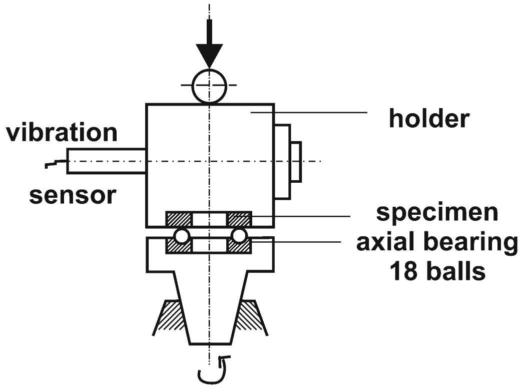


Fig. 1. Principle of Axmat equipment.

3. Results

The CrL type material used in this paper has in the case of 0.3 % C a ferrite + pearlite structure, while in the other case (0.7 % C) it consists mostly of a very fine pearlite. Both structures correspond to the given chemical composition and to cooling rate after sintering ($\sim 0.3\ \mathrm{K\,s^{-1}}$). It is in agreement with observation in literature and with CCT diagram for this type of steels as well [12]. Plasma nitriding, similarly to the classical one, is expected to create a layer on the surface of the material, which would have high hardness, and would have specific properties that enable it to withstand conditions in which it has to perform. Similarly to gas nitriding, apart from the nitrided layer, an ε-layer is formed, the thickness of which in all researched variants was approximately 10 µm – Fig. 2. The layer has a relatively high porosity, which – as presented further – can have a negative influence on contact fatigue. Its hardness, particularly in the CrL + 0.3C material, is lower than the next, nitrided layer, as shown in Fig. 3.

Microhardness profile on the cross section is presented in Fig. 3a,b. If the value of 500 HV0.05 is considered to be the minimum hardness valid for the nitrided surface, then we can see that the effective depth of nitriding is around 150–200 µm for the CrL + 0.3C material and around 125–150 µm for the CrL + 0.7C. The effective depth can also be determined by fracture.

The hard nitrided layer at classical impact loading breaks in brittle fashion, which results in cleavage damages – Fig. 4a. Figure 4b shows detail of cleavage facets on the brittle fracture surface suffered by this layer.

The hardness of the HV10, in terms of the ε-layer, could not be measured exactly as this layer was very thin and the measuring device did not allow a precise setting of the indenter into the centre of this layer which caused a large scatter in the measurements.

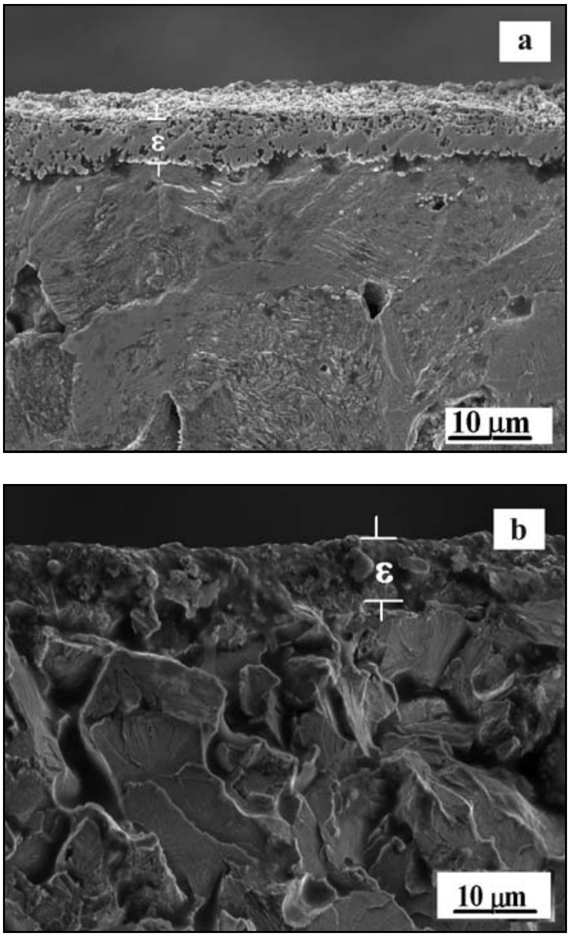


Fig. 2. ε-layer on surface of material CrL + 0.3C (LOM) (a) and CrL + 0.3C (SEM) (b).

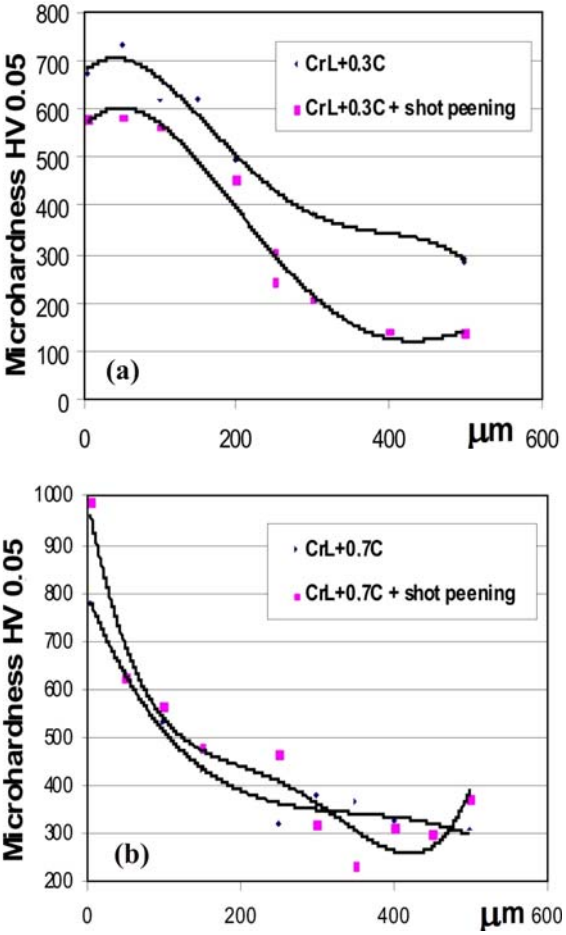


Fig. 3. Microhardness versus depth under surface in CrL + 0.3C (a) and CrL + 0.7C (b).

Table 1. Values of hardness and microhardness of CrL material (Pn – plasma nitriding, Sp – shot peening)

	0.3C Pn	0.3C Pn + Sp	0.7C Pn	0.7C Pn + Sp
HV10	338	453	428	508
HRB	85	95	93	98
HV0.05	600	725	785	980

However, it is surprising that the ε-layer did not suffer brittle damage. Its damage cannot be unambiguously described by any of the known fractographic features – Fig. 2b. Surface hardness, the decisive factor affecting the contact fatigue properties, is summed up in Table 1.

Measuring hardness of HV10 is rather questionable. After nitridation the surface is totally black and no impression can be seen. It is necessary to grind the surface slightly, thereby removing part or the entire ε-layer. This setback was to be eliminated by measuring the HRB. However, these results do not reflect the direct proportion existing between them and the life cycle fatigue, either.

Tests of contact fatigue have produced results as in Fig. 5a,b. As it follows from the graphs, with the CrL + 0.3C material, the stress values obtained were 1090 or 1800 MPa, and with the CrL + 0.7C material they were 1530 or 2000 MPa. In order to separately compare the effects of nitriding and shot peening, one has also to take into account the values for the purely sintered status. These values are presented in [10]. Summary of the results is shown in Table 2.

The results obtained are consistent with the material behaviour. Softer materials, following shot peening, have a higher percentage of service life increment than the harder ones. For CrL + 0.3C the nitridation itself increased its fatigue limit by 65 %, and for the materials with 0.7C only by 38 %.

In the case of combination of shot peening and nitridation, the fatigue limit of the CrL + 0.3C material increased by 172 % when compared with the as-sintered state. In the variant with 0.7 % C it was

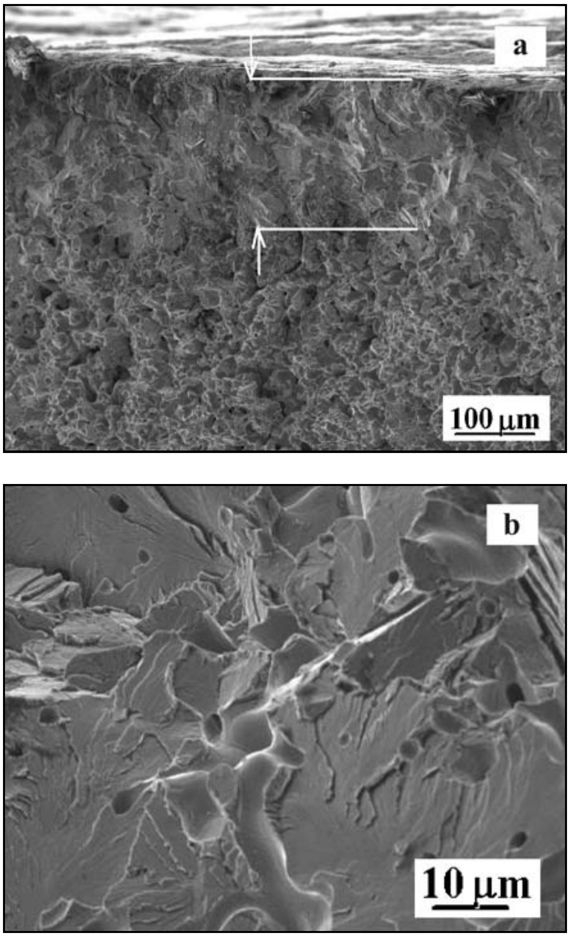


Fig. 4. Brittle fracture of nitride layer (a) and cleavage facette of nitride layer (b).

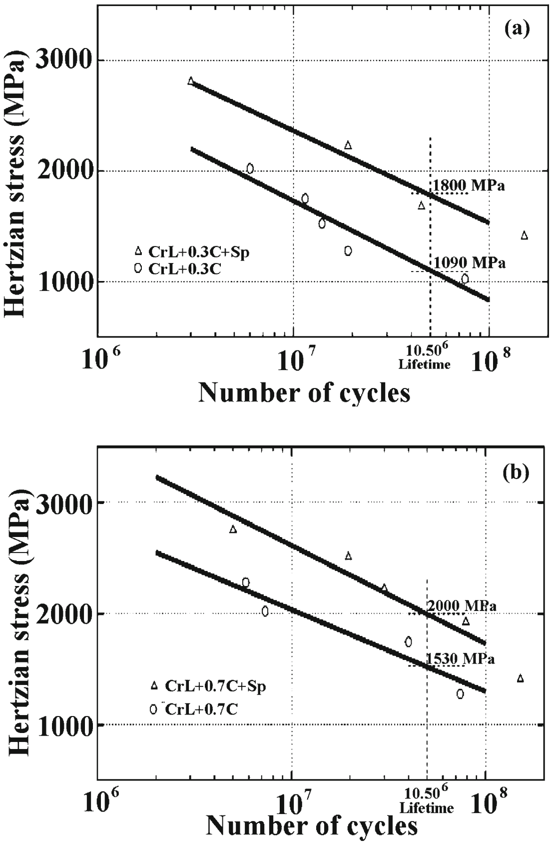


Fig. 5a,b. Life cycle. Contact fatigue curve.

Table 2. Type of treatment versus CF limits

	CrL + 0.3C (MPa)	(%)	CrL + 0.7C (MPa)	(%)
Sintered	660	100	963	100
Shot peening	720	109	1000	103
Plasma nitriding	1090	165	1530	138
Sp + Pn	1800	272	2000	207

only by 107 %. Absolute values are naturally higher for materials with higher initial hardness.

Metallographic observation has revealed that pittings are formed by gritting out material from cracks that originated on the surface – Fig. 6. Here, around the pitting, one can see a whole range of small cracks generated by their interlinking. This is supported in Fig. 7, where the final cracks evidently come from the cracks in the ε-layer and their subsequent interlinking. Primary causes that generate cracks can be attributed to two factors. The first influence are the pores in the ε-layer, see Fig. 2, which have nothing in common with the real porosity of materials, however. It is not known, why this layer is so porous after plasma nitriding, and the solution of this problem falls beyond the scope of this work. The second one is the presence of complex oxide particles, which caused the layer to burst, and develop real cracks. The nature of these particles (complex oxides of Fe-Cr-Al-O type) was confirmed by EDAX analysis – Fig. 8a,b. The origin of Zn lies in the use of zinc stearate as a lubricant, which serves to protect dies against abrasion wear. These results are in conformity with the ones achieved in [13, 14], and what is more, the material used in the research was from the same batch as in our case.

The process of pitting formation, from its initiation, through growth up to reaching sizes and numbers that cause the testing device to switch off, is another phenomenon requiring certain attention. The experimental materials were loaded so that the Hertzian stresses reached 2000 MPa.

The pre-marked points on the sample surface were microscopically monitored in certain time intervals (~ 30 min) and the pittings generated were subsequently measured. This enabled development of the graphical relationship shown in Fig. 9.

In samples with shot peened and subsequently

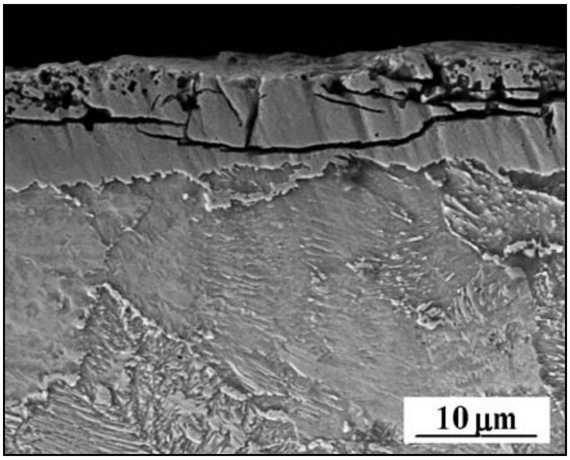


Fig. 6. Surface cracks on the orbit CrL + 0.3C.

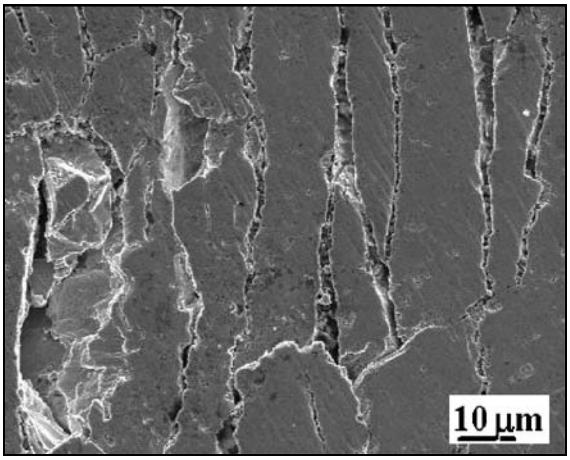
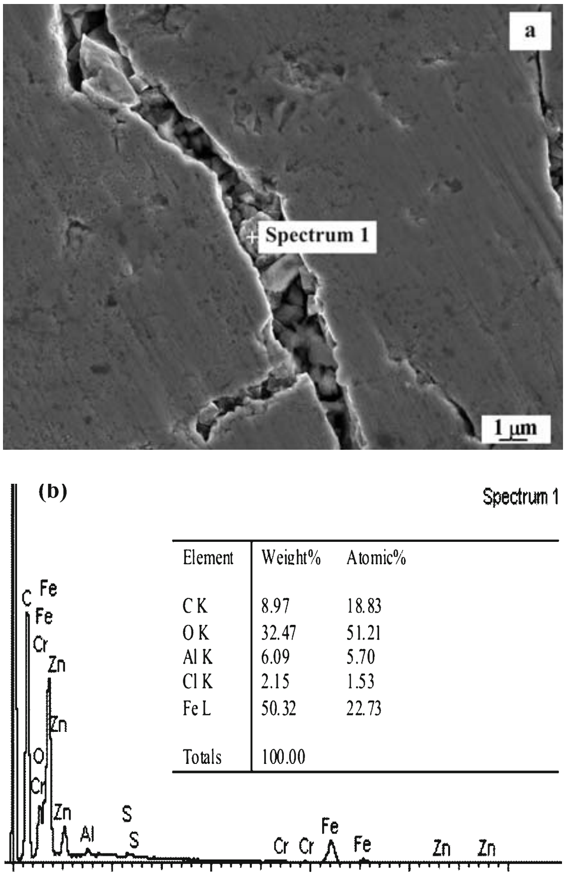


Fig. 7. Cracks in the ε-layer CrL + 0.3C.



Element	Weight%	Atomic%
C K	8.97	18.83
O K	32.47	51.21
Al K	6.09	5.70
Cl K	2.15	1.53
Fe L	50.32	22.73
Totals	100.00	

Fig. 8a,b. SEM analysis of particles causing cracking of the surface layer CrL + 0.3C.

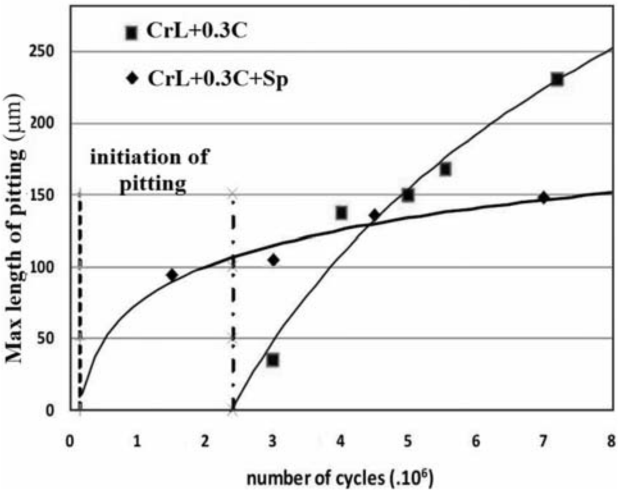


Fig. 9. Size of the pittings versus number of cycles.

plasma nitrided surfaces small pittings develop already at 240 thousand cycles. The value is representing a limit below which formation of pittings is invisible. Despite this fact, in nitrided samples without shot peening pittings become visible only at values as high as 2.4 million cycles. Upon exceeding this limit, the growth of pittings is faster here than in the shot peened samples.

Earlier initiation of pittings in the shot peened materials is probably related to the exhausted deformation capability of the material. The material no longer accepts further loading under the contact stress, and at the interface between the matrix and the hard, secondary particles, cracks are developing, becoming interlinked and pittings are formed. Similar behaviour can also be expected in the samples with 0.7 % C. Paradoxically, even if the formation of pittings starts earlier in the shot peened samples than in the other ones, the nature of this process causes that their growth in the size and number sufficient to terminate testing (switching off the testing equipment) is slower, and takes longer time than it is in the case

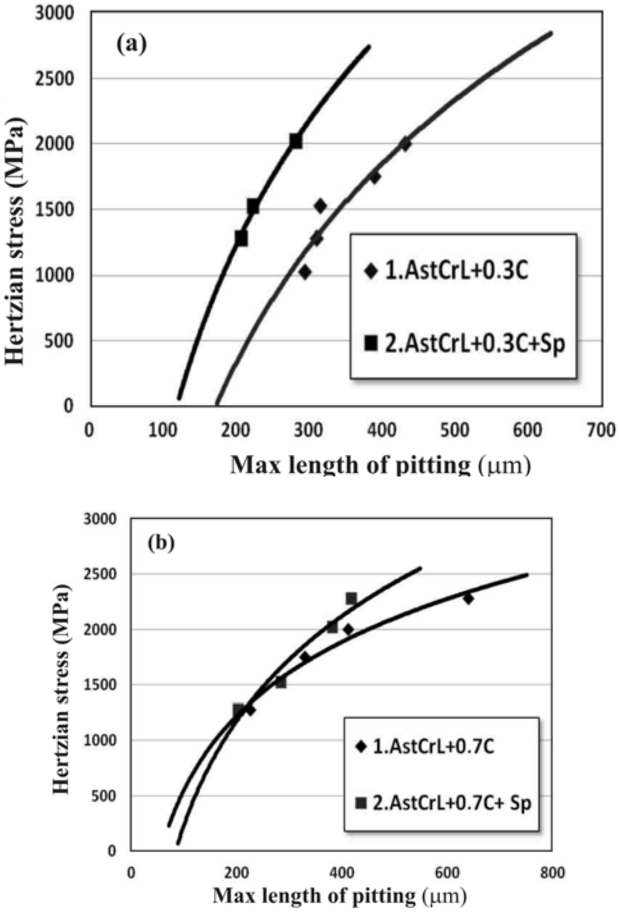


Fig. 10a,b. Relationship between the size of the pittings and the Hertzian stress and the kind of material.

of samples without shot peening.

Logarithmic growth of the size of pittings, except for the number of cycles run can also be related to the magnitude of the applied stress. It is visible in presenting the graphical relationships between the stress and the corresponding size of the pitting after stopping the testing equipment. These graphical relationships are seen in Fig. 10a,b.

The observed relationships show smaller sizes of pittings in shot peened materials, as well as the fact that they grow more slowly than in the case of ones without shot peening. This reasoning is valid when the Hertzian stress is used.

4. Conclusions

1. Surface hardening of the investigated sintered materials by shot peening as well as plasma nitriding improved the resistance to contact fatigue. It indicates that the use of both technologies has a synergic effect. If the actual shot peening will improve the contact fatigue resistance by 9 % and the actual nitridation by 65 %, their combination, i.e. plasma nitriding preceded by deformation hardening by shot peening, can achieve much higher improvement – in this case it was an increase of 172 % (Table 2).
2. Higher values of fatigue resistance are demonstrated by materials with higher carbon content, with correspondingly higher values of hardness.
3. Even if the material hardness is the determining factor for the contact fatigue resistance, it seems that a similarly important role is played by the material structure itself. The influence of hard particles found either on or slightly below the surface is highly unfavourable. In our case, it is above all the complex oxides that initiate cracks on the surface.
4. Pittings, results of the contact fatigue, are more likely to start in shot peened materials, but then they develop more slowly than the pittings in materials without shot peening.

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