# The effect of manganese phosphate coating treatment on fretting fatigue strength of AISI 1045 steel

Y. Totik\*, A. Caglaroglu, R. Sadeler

Department of Mechanical Engineering, Faculty of Engineering, Ataturk University, 25240 Erzurum, Turkey

Received 10 April 2007, received in revised form 23 May 2007, accepted 28 May 2007

### Abstract

The effects on fretting fatigue strength of manganese phosphate coated and uncoated AISI 1045 steel were investigated in this study. The results showed that the fretting fatigue limit of phosphate coated specimens was higher compared with uncoated specimens since the manganese phosphate layer occurring on surface decreased the friction force and friction coefficient in contact region. However, the plain fatigue limit did not increase because the surface compressive residual stress did not occur on the surface and the hardness did not change. The phosphate coating treatment delayed the oxidization of particles separated from contact region, which resulted in the increase of the fretting fatigue limit.

Key words: phosphate coating, plain fatigue, fretting fatigue

#### 1. Introduction

Fretting is a special wear process that concerns materials in contact that are designed to be fixed but undergo small relative movements (less than about 0.003 inches) due to cyclic loads [1]. When two pieces of material, contacted together by a load, are subjected to a transverse cyclic loading, so that one contacting face is relatively displaced cyclically parallel to the other face, in the presence of high contact stress, wear on the mating surfaces occurs. The destructive process that reduces the fatigue properties by promoting early crack initiation is usually mentioned as fretting fatigue. It is commonly observed in several practical situations, such as riveted and bolted joints, disk and blade attachment (dovetail joints) in gas turbine and jet engines [2–7].

Fretting fatigue damage has great safety and economical influence on the industry. During fretting the fatigue strength decreases to less than one-third of that without fretting. The fatigue strength decreases and the maintenance cost of components in real applications raises owing to contact pressure and tangential stress at the contact area, where fretting fatigue cracks initiate and propagate. The variables effecting as directly or indirectly the fretting behaviour are: applied load, contact load, relative slip, contact geometry, coefficient of friction and surface conditions. In general, there are two methods to improve fatigue behaviour of machine components under fretting conditions: mechanical treatment of surface and coating of surface. Surface coatings may prevent the contact region from the fretting phenomenon and may be separated into two types as hard and soft. Soft surface coatings are used to reduce the fretting wear and the fretting fatigue failure [8–10]. Since coatings could reduce the coefficient of friction and behave as a self-lubricant, they improve fretting fatigue behaviour.

Phosphate coating is a surface treatment applied to cast iron, steel or steel based substrates with a solution, whereby the surface of the metal is converted to an integral, mildly protective layer of insoluble crystalline [11–13]. It is a process used to produce a crystalline, oil absorbing coating with an excellent wear resistance. Phosphate coatings are utilized in a variety of industrial markets to include: automotive, aerospace, military and nuclear energy [14, 15]. These coatings are applied to facilitate initial running of in new parts, such as piston rings, camshafts, cylinder liners, differential gears and transmission gears. The etch pattern produced by manganese phosphate on

<sup>\*</sup>Corresponding author: tel.: +90 442 2314858; fax: +90 442 2360957; e-mail addresses:  $\underline{ytotik@atauni.edu.tr}$ ,  $\underline{yasrtotik@yahoo.com}$ 

Table 1. Chemical composition of the AISI 1045 steel

Element	С	Si	Mn	Р	S	$\mathbf{Cr}$	Mo	Ni	
wt.%	0.4220	0.2440	0.6070	0.0190	0.0330	0.1970	0.0332	0.1220	

Table 2. Parameters of manganese phosphate coating

Free acid point (FS)	Total acid point (GS)	$ \begin{array}{c} \text{Iron content} \\ (\text{mg } l^{-1}) \end{array} $	$\begin{array}{c} \text{Temperature} \\ (\ ^{\circ}\text{C}) \end{array}$	Dipping time (min)	
10.8	114.6	0.3	90	15	

the surface of parts greatly improves oil retention and prevents galling of contact surfaces [16].

The objective of this study is to investigate the effect of manganese phosphate coating treatment on fretting fatigue strength in contact with similar material under contact pressures of AISI 1045 steel.

## 2. Experimental procedures

The chemical composition of AISI 1045 steel used in this study is given in Table 1. The specimens and pads used in the experiments were machined with a CNC machine from cold drawn cylindrical material with a 12 mm diameter. Two types of specimens were used to determine the plain fatigue and fretting fatigue strength. The fretting fatigue specimens were machined to produce two parallel flats, reducing the thickness to 5 mm as shown in Fig. 1a. For plane fatigue tests, geometrical dimensions of specimens were given in Fig. 1b. The contact pads were machined from the same material as the fatigue specimens as shown in Fig. 1c. The plain and fretting fatigue test specimens and pads were properly polished with alumina paste (0.3  $\mu$ m) to produce smooth surface after wet polishing with silicon carbide emery papers grit 400, 600, 800 and 1200.

The hardness of the specimens before and after the manganese phosphate coating was measured as Vickers hardness using a PC-controlled Buehler-Omnimet tester. In addition, the surfaces roughness before and after the manganese phosphate coating was evaluated by using a Mitutuyo profilometer.

The suitable coating parameters are usually determined as the type and microstructure of the materials and the characteristics of the expected surface quality. Therefore, the surface of AISI 1045 steel specimens was degreased in 12 % NaOH solution to clean out greases and exposed to an acid pickling to send away all surface oxides and other dirt before applying coating treatments. Then, the phosphate coating treatment was applied to the specimens as immersion in a phosphate coating bath at a temperature of 90 °C,



Fig. 1. Shape and dimensions of a) the fretting fatigue specimen and b) the plain fatigue specimen and c) the contact pad.

and the parameters of phosphate coatings are given in Table 2.

The plain fatigue tests were carried out until the complete failure of specimens using a rotating bending fatigue machine (SATEC) with R = -1 at room temperature in air and a rotational speed of 5000 rpm. The fretting fatigue tests were carried out using the same machine under the same parameters as for the plain fatigue tests for the fretting fatigue specimens with a flat area of 3.5 mm width and 24 mm length, for which other geometrical dimensions were the same as that of the plain fatigue test specimen.



Fig. 2. Schematic drawing of the fretting fatigue testing system at the rotating bending machine.

The rotating bending fretting test setup employed in the present study is schematically shown in Fig. 2. The fretting fatigue test specimen and bridge pad were contacted in the contact pressure of 100 MPa. The contact pressure between the surface of the specimens and bridge pads was controlled by an adjusting screw with a torque driver. The fatigue limit was accepted as the stress values corresponding to  $3 \times 10^6$  cycles, and fatigue test machine was shut down when this value was reached. The *S-N* curve and fatigue limit were obtained using the standard JSME S 002 and staircase method, respectively. Six uncoated and manganese phosphate coated specimens were used for the *S-N* curves with 50 % probability.

The surface micrographs of the coatings, fracture surfaces, pad contact areas on the surfaces and contact surfaces of the pads were characterized using a Jeol 6400 Scanning Electron Microscope (SEM).

#### 3. Results and discussion

The hardness of the specimens after phosphate coating treatment was about 222 HV while the hardness of the specimens before coating treatment was about 216 HV. The manganese phosphate layer formed on the steel surface by the chemical reaction with phosphate crystals was softer than the material surface. Therefore, it was observed that the hardness value did not provide a considerable increase.

The surfaces roughness before and after the manganese phosphate coating was determined using a Mitutuyo profilometer. After the manganese phosphate coating this value increases to Ra = 0.60 while it was about Ra = 0.40 before the coating. It was observed that manganese phosphate coating treatment caused a little increase in the surface roughness. The beneficial effect of surface roughness in enhancing fretting fatigue limit has been reported in literature [17, 18].



Fig. 3. S-N curves obtained from plain fatigue tests of the uncoated and coated AISI 1045 steel.

The results of plain fatigue and fretting fatigue tests conducted on the uncoated and coated specimens given as S-N curves are shown in Figs. 3–5. It is observed that the phosphate coating treatment does not influence fatigue limit of the plain fatigue specimens as shown in Fig. 3. The fatigue limit is 334 MPa for the case of the coated specimens while it is 332 MPa for the case of the uncoated specimens from the plain fatigue tests. It is clear that plain fatigue limits are almost the same. In other words, there is no significant effect of the coating on the fatigue limit of the plain fatigue specimens as a significant increase in the surface hardness after coating does not take place as earlier mentioned in text. Besides, it is shown that the crack initiation in plain fatigue accounts for a large portion of the fatigue life and occurs through the movement of dislocation in the surface region by cyclic shear stress [19].

250 Uncoated Fretting Fatigue 4 4.5 5 5.5 6 6.5 7 7.5 Log N

Uncoated Plain Fatigue

Fig. 4. S-N curves obtained from plain fatigue and fretting fatigue tests of the uncoated AISI 1045 steel.



Figure 4 shows the S-N curves obtained from the plain fatigue tests and fretting fatigue tests for the uncoated specimens. From the figure it is seen that the fatigue limit reduces with fretting phenomenon. The fatigue limits for the plain fatigue and fretting fatigue for the uncoated AISI 1045 steel are found around 332 and 282 MPa, respectively. It is apparent that fretting has a harmful effect on fatigue limits. The fatigue cracks initiated from the contact region due to frictional shear stress concentration locally introduced by fretting, and multiple crack initiation sites were observed in the contact region in fretting phenomenon. Therefore, the decrease in the fatigue limit by the fretting phenomenon is considered due to the decrease in crack initiation duration caused by the local stress concentration, and the acceleration of the initial crack propagation by fretting [20]. One of the main mechanisms is regarded as the wedge effect where the wear debris goes into the fretting fatigue crack [21]. However, after the crack is filled with the wear debris, this effect decreases since the wear debris cannot go into the crack. On the other hand, fretting fatigue strength is higher than the plain fatigue strength in the low cycle fatigue life region, which indicates that the applied stresses affect the plain and fretting fatigue strength.

Figure 5 shows the S-N curves obtained from the fretting fatigue tests for the uncoated and coated specimens. Despite the fact that the manganese phosphate coating treatment has not great effect on the fatigue limit of the plain fatigue specimens, it has had a positive effect on the fatigue limit of the fretting fatigue specimens. The fatigue limit of the manganese phosphate coated fretting fatigue specimens is higher than that of the uncoated fretting fatigue specimens. The fretting fatigue limit of the coated specimens (298) MPa) is higher than the uncoated specimens (282) MPa). This improvement could be explained as follows: The manganese phosphate layer occurring on the surface increased crack initiation life due to the local stress concentration caused by fretting and decreased the acceleration of the initial crack propagation, and improved the ease of sliding and the reduction of associated wear of two steel surfaces sliding one against the other. The phosphate crystals formed on the surface are dense and fine as shown in Fig. 6, which provides a protective layer against wear of the surface. Thus, the layer occurring on the material surface facilitates the sliding between material surfaces, and reduces wear.

Typical SEM images of the crack surface of the uncoated and coated specimens after the plain fatigue tests are shown in Fig. 7. The crack forms in three stages in fatigue known as: crack initiation, propagation and fracture. The plastic deformation has an important role in both crack initiation and propagation stages. In the uncoated specimens, the plain fatigue cracks initiate at the surface and propagate for the plain fatigue tests as shown in Fig. 7a, and most of the fatigue life is spent in crack initiation. As seen from Fig. 7b, similar behaviour is observed for the coated specimens, and the crack initiates at the surface of the coating and propagates through the interface into the substrate. Since compressive residual stresses are not present at the surface of the coating, and the hardness of the coating is not higher than that of the substrate, the crack initiation resistance is about the same compared to



Alternatting Stress (MPa)

430

400

370

340

310

280

430

400

370

340

310

280

Alternatting Stress (MPa)



Fig. 6. SEM image of the manganese phosphate layer on surfaces of AISI 1045 steel.



Fig. 7. SEM images of the fracture surfaces a) uncoated and b) coated specimens tested under plain fatigue loading.

the uncoated substrate material for the plain fatigue tests.

Figure 8a,b shows the fracture surfaces of the man-



Fig. 8a,b. Fracture surfaces of coated fretting fatigue specimens for different magnification.

ganese phosphate coated fretting fatigue specimens. The fretting fatigue crack initiates from the surface of the fretting specimen at the contact region with the pad (the boundary between slip and stick regions) and then propagates through the interface into the substrate. In fretting fatigue tests, multiple crack initiation sites are observed in the contact region. The crack growth takes place on the fracture surface under the fretting as shown in Fig. 8 from which it can be seen that the crack growth zone under the contact pressure on the fracture surface becomes smaller. As the friction coefficient of the coating is lower than that of the substrate, the crack initiation resistance is higher compared with the uncoated substrate material.

Figure 9 includes the SEM images of fretting scars forming on the substrate surfaces during fretting fatigue tests. The scar and fracture area occurring on the surface after the fretting fatigue tests are shown in Figs. 9a and 9b for the uncoated and coated specimens, respectively. In addition, Fig. 10 shows the schematic illustration of the fretting fatigue crack initiation and progress. Stick-slip status takes place dur-



Fig. 9. SEM images of wear scar of a) uncoated fretting fatigue specimen and b) coated fretting fatigue specimen in fretted area.



Fig. 10. Schematic illustration of fretting damage; h – depth of fracture initiation zone site [23].

ing fretting fatigue tests (the centre (stick) and edge (slip) of the contact area) and the slip and stick re-



Fig. 11. SEM images of pad surfaces in contact regions for a) uncoated specimen and b) coated specimen.

gions are unclear at the coated fretting specimens as shown in Fig. 9b. The abrasive wear forms in the slip area due to fretting condition and the debris occurring during abrasive wear is swept out from the contact region. The contact load of the pad is higher than the total contact load applied to the slip region, and the total contact pressure acting in the slip region is lower than that in the stick region. Also stress concentration occurs near the boundaries between the stick and slip regions, and the crack could be easily initiated [19, 20, 22]. The crack at the uncoated specimens initiates between the slip and stick regions of the contact area due to the stress concentration effect caused by fretting as shown in Fig. 9a. The main crack in the coated fretting specimens initiates in a region outside the stick and slip region as shown in Fig. 9b since the coating treatment reduces the contact pressure.

Phosphating is a widely used method of reducing wear on machine elements and moving parts [23]. The manganese phosphate coatings, supplemented with proper lubricants are most commonly used for wear resistance applications. The wear surfaces in contact region of bridge-type pads used as contrary components are shown in Fig. 11. The wear at each pad takes place as shown in Fig. 11, but much wear quantity is observed in pads during contact with the uncoated specimens. Therefore it was found that the friction coefficient and wear quantity in pad surfaces decreased with the manganese phosphate. The manganese phosphate layer formed on the steel surface by chemical reaction with phosphate crystals is softer than the material surface, and supports effective running-in, and the phosphate treatment can reduce the formation of wear scars [24].

# 4. Conclusions

The following conclusions are drawn from the study of the fretting fatigue of the manganese phosphate coated and uncoated AISI 1045 steel, with the same contact material, under the contact pressures of 100 MPa.

- The fretting phenomenon reduces the fatigue limit of the uncoated and coated specimens to a large extent because of the decrease in crack initiation life caused by the local stress concentration due to fretting.

- The plain fatigue limit is not enhanced by the manganese phosphate coating treatment since the surface compressive residual stress does not occur on the surface, and the hardness value does not increase.

- The fretting fatigue limit of the coated specimens is higher compared with the uncoated specimens since the manganese phosphate layer occurring on surface increases crack initiation life caused by the stress concentration due to fretting and decreases the acceleration of the initial crack propagation, the friction force and friction coefficient between contact surfaces.

– After studying SEM images, fine and dense phosphate crystals were observed on the surface. They improve the sliding properties of the surfaces, reduce wear and thus increase the fretting fatigue limit. In addition, phosphate coating treatment prevents the oxidization of particles separated from contact region, which results in the increase of the fretting fatigue limit.

#### Acknowledgements

This research was supported by Ataturk University under the Project number of BAP 2002/40, which is gratefully acknowledged by the authors.

#### References

- WATERHOUSE, R. B.: Fretting Corrosion. Oxford, Pergamon Press 1972.
- [2] WATERHOUSE, R. B.: Fretting Fatigue. London, Applied Science Publishers 1981.
- [3] LEE, H.—MALL, S.—SATHISH, S.—BLODGETT, M. P.: Materials Letters, 60, 2006, p. 2222.
- [4] BRYGGMAN, U.—SODERBERG, S.: Wear, 125, 1988, p. 39.
- [5] FOUVRY, S.—KAPSA, P.—VINCENT, L.: Wear, 200, 1996, p. 186.
- [6] NIX, K. J.—LINDLEY, T. C.: Fract. Eng.Mater. Struct., 8, 1985, p. 143.
- [7] HUTSON, A. L.—NICHOLAS, T.—GOODMAN, R.: International Journal of Fatigue, 21, 1999, p. 663.
- [8] JAHANMIR, S.—ABRAHAMSON, E. P.—SUH, N. P.: Wear, 40, 1976, p. 75.
- [9] VADIRAJ, A.—KAMARAJ, M.: Surface and Coatings Technology, 200, 2006, p. 4538.
- [10] LEE, H.—MALL, S.—ALLEN, W. Y.: Materials Science and Engineering A, 420, 2006, p. 72.
- [11] TOTIK, Y.: Surface and Coatings Technology, 200, 2006, p. 2711.
- [12] WENG, D.—JOKIEL, P.—UEBLIS, A.—BOEHNI, H.: Surface and Coatings Technology, 88, 1996, p. 147.
- [13] UEBLIS, A.—WENG, D.—BOEHNI, H.: In: Proceedings of the First Swiss Conference on Materials Research for Engineering Systems. Ed.: Ilschner, B. et al. Wabern, Technische Rundschau 1994, p. 314, ISBN 3444104367.
- [14] LORIN, G.: Phosphating of Metals. Middlesex, Finishing Publications 1974.
- [15] RAUSCH, W.: The Phosphating of Metals. Middlesex, ASM International Metals Park, Finishing Publications 1990.
- [16] FREEMAN, D. B.: Phosphating and Metal Pre-Treatment – A Guide to Modern Processes and Practice. New York, Industrial Press 1986.
- [17] WATERHOUSE, R. B.—LINDLEY, T. C. (Eds.): Fretting Fatigue, ESIS, vol. 18, London, Mechanical Engineering Publications 1994, p. 339.
- [18] LI, C. X.—SUN, Y.—BELL, T.: Mater. Sci. Technolgy, 6, 2000, p. 1067.
- [19] SADELER, A.—SENGUL, A. B.: Kovove Mater., 44, 2006, p. 235.
- [20] SUMITA, H.—NAKAZAWA, K.—HAMANO, R.— MARUYAMA, N.—ABE, Y.—NISHIJIMA, S.: Report of the National Research Institute for Metals, No. 14, 1993, p. 207.
- [21] ANTONIOU, R. A.—RADTKE, T. C.: Mater. Sci. Eng. A, A237, 1997, p. 229.
- [22] TAKEDA, J.—NIINOMI, M.—AKAHORI, T.—GU-NAWARMAN, M.: International Journal of Fatigue, 26, 2004, p. 1003.
- [23] PLACEK, D. G.—SHANKWALKAR, S. G.: Wear, 173, 1994, p. 207.
- [24] Federal-Mogul Powertrain Systems, Federal Mogul, Burcheid, Germany, ISO 6621-4, 1998, p. 32.