The Significance and Determination by Image Analysis of Microcrack Density in Hard Chromium Plating

Marcelino Pereira do Nascimento^{*} & Herman Jacobus Cornelis Voorwald Fatigue and Aeronautical Material Research Group State University of São Paulo, Guaratinguetá City, São Paulo State, Brazil

It is well known that the microcrack density is a fundamental parameter in hard chromium electroplating. The chemical and mechanical properties of this coating are widely dependent on its microcrack density. In this paper a simple image analysis procedure to determine microcrack density is presented in order to demonstrate it as a fundamental tool to estimate the fatigue, corrosion and wear behavior, as well as the residual stress field of a coated component. For this purpose, the image analysis procedure was carried out on two kinds of hard chromium plating - one called "accelerated" (high velocity of deposition and fluoride-free) and the other conventional (with fluoride). The coatings were applied on samples of AISI 4340 aeronautical steel, which is widely used in aircraft landing gear components. To characterize the practical significance of this study, the microcrack density results were related to the fatigue, wear and corrosion behavior from previous study and to the residual stress field in the coatings.

Keywords: microcrack density, image analysis, hard chromium electroplating, surface coating, stereology.

Introduction

Despite pressures to identify alternatives to conventional hard chromium plating related mainly to environmental requirements,¹ studies to improve its properties^{2,3} have increased in recent years in order to maintain its traditional applications. For both decorative and functional applications, chromium electroplating exhibits excellent wear, abrasion and corrosion resistance properties. However, it is well known that hard chromium plating reduces the fatigue strength of mechanical components. This is mainly attributed to high tensile internal residual stresses and microcrack density inherent in the coating.⁴⁻⁶ As an example of recent research, high-efficiency and fluoride-free hard chromium electroplating (in this paper called "accelerated") are important candidates for replacing conventional hard chromium.

The term "hard chromium" is used to indicate electrodeposits with thicknesses above 2.5 μ m.⁶ For functional applications in particular, hard chromium is a well-established process in the aeronautical, automotive and petrochemical fields.⁷ In the aeronautical industry, this coating is applied on several aircraft landing gear components such as shock absorbers, hydraulic cylinders and shafts. Figure 1 shows some hard chromium plated components of an aircraft landing gear fabricated by the Brazilian Aircraft Industry (Embraer Liebherr/EDE).

Microcracks in hard chromium plating consist of equiaxial superficial crack networks, as well as cracks that do not extend through the thickness of the coating. They arise during the electroplating process when the thickness reaches over 0.5 μ m and the internal tensile stress exceeds the cohesive strength of chromium.^{6,8} These high internal tensile stresses are produced by the decomposition of chromium hydrides, and by the change of the crystalline microstructure of the remaining chromium hydrides from HCP to BCC, resulting in over a 15% volume reduction of the coating.⁸ The high stresses and consequently the microcrack density are a function of the bath composition, temperature and current density.⁹



Figure 1-Aircraft landing gear; hard chromium electroplated components (Courtesy of Embraer Liebherr -EDE/BR).

Marcelino Pereira do Nascimento Fatigue and Aeronautical Material Research Group State University of São Paulo - UNESP 333, Ariberto Pereira da Cunha Ave.

Guaratinguetá City – CEP: 12516-460

São Paulo State, Brazil.

Phone: 55-12-3123-2865

Fax: 55–12-3123-2852

^{*} Corresponding author

E-mail: marcelino.nascimento@gmail.com

Basically, the mechanical and chemical properties of the chromium deposits are widely dependent on their microcrack density. In previous studies,³⁻⁵ the influence of microcrack density on hard chromium plated AISI 4340 aeronautical steel was verified, in terms of fatigue, corrosion and wear resistance. Therefore, the microcrack density is a very important factor in controlling both the chemical and mechanical properties of the coating and consequently its measurement is fundamentally important. The aim of this study is to employ a simple image analysis procedure to determine the microcrack density in hard chromium electroplating, in order to demonstrate its importance as a powerful tool for optimizing the coating process parameters. Our other objective is to estimate the final mechanical and chemical properties required for specific industrial applications.

Experimental procedure

Hard chromium plating

Conventional hard chromium electroplating was carried out from a solution containing 250 g/L of CrO_3 and 2.5 g/L of H_2SO_4 , at 50 to 55°C, at a current density ranging from 31 to 46 A/dm², and a deposition rate of 25 μ m/hr. A bath catalyst based on sulfate and fluoride was used.

The "accelerated" hard chromium electroplating was carried out from a solution with 250 g/L of CrO_3 and 2.7 g/L of H_2SO_4 , at 55 to 60°C, with a current density from 55 to 65 A/dm², and a deposition rate of 80 µm/hr. A bath catalyst based on sulfate and without fluoride was used. Both coatings were evaluated in accordance with Brazilian Aircraft Industry (Embraer-Liebherr/EDE) specifications.

Procedures for microcrack density determination and image analysis

For the microcrack density determination, samples were prepared from normalized AISI 4340 steel (widely employed in aircraft landing gear components in the quenched and tempered condition), with 1 mm thickness \times 25 mm \times 25 mm and superficial roughness $R_a \approx 0.2 \ \mu m.$

The samples were then either plated with accelerated or conventional hard chromium, both a 100 μ m thickness. The surface microcracks were enhanced through reverse-current etching at 25

A/dm² for 30 sec in each chromium bath. After that the samples were analyzed by means of a Nikon/Apophot optical microscope with a SPOT Insight QE digital camera. Images were obtained of 20 different positions of each sample with magnifications of 100× and 200× and then calibrated as shown in Figs. 2 and 3.

The microcrack density was quantitatively determined with Image Pro-Plus 4.0 and Material Pro-Analyser 3.1 software. In order to improve the contrast and remove the background, a green filter was applied on the captured images. Afterward, the images were fitted by linear equalization and the intercept pattern was applied (Fig. 4). A system containing 20 test lines was superimposed on the images (Fig. 5). Furthermore, a histogram (Figs. 6 and 7) was employed in order to improve the adjustment of the image to the intercept patterns, and the quantification of the number of interceptions of the surface microcrack network with a grid line was then performed.

A simple formula to quantify the microcrack density was used, as follows:¹⁰

$$Microcrack Density = N_i / L_i$$
(1)

where:

 L_t = total length of the grid line and

 N_i = the total number of line intersections with the microcrack network.

Results

Importance and determination of microcrack density in hard chromium electroplating

Table 1 presents the microcrack density values obtained for 20 positions in both the conventional and accelerated hard chromium electroplated samples. The results indicate an average of 1512 cracks/cm and a standard deviation of 190.6 cracks/cm for the "accelerated" hard chromium compared to an average of 223 cracks/cm and a standard deviation of 57.5 cracks/cm for the conventional deposit.

The results are in accordance with the literature^{6,11} and can explain the better corrosion and wear resistance performance and the poor fatigue strength of the accelerated hard chromium plating, which will be discussed later.



Figure 2-Accelerated hard chromium superficial cracks.



Figure 3-Conventional hard chromium superficial cracks.

Journal of Applied Surface Finishing



Figure 4—Applications of the intercept pattern.



Figure 5-Applications of grid line test.

Journal of Applied Surface Finishing



Figure 6-Partial adjustment of the histogram.



 $Figure \ 7-Complete \ adjustment \ of \ the \ histogram.$

Conventional Hard Chromium			Accelerated Hard Chromium		
Length of test line, $L_t = 1.2400$ cm			Length of test line, $L_t = 0.2030$ cm		
Sample	(<i>N_i</i>) # Intercepts	$\frac{N_i/L_t}{(\text{cracks/cm})}$	Sample	(<i>N_i</i>) # Intercepts	$\frac{N_i / L_t}{(\text{cracks/cm})}$
100 C1	399	321.774	200 A1	260	1280.788
100 C2	280	225.806	200 A2	257	1266.01
100 C3	185	149.194	200 A3	277	1364.532
100 C4	186	150	200 A4	296	1458.128
100 C5	236	190.322	200 A5	316	1556.65
100 C6	375	302.419	200 A6	349	1719.212
100 C7	336	270.968	200 A7	313	1541.872
100 C8	309	249.194	200 A8	325	1600.985
100 C9	377	304.032	200 A9	277	1364.532
100 C10	320	258.064	200 A10	286	1408.867
100 C11	167	134.677	200 A11	304	1497.537
100 C12	219	176.613	200 A12	243	1197.044
100 C13	359	289.516	200 A13	303	1492.612
100 C14	195	157.258	200 A14	279	1374.384
100 C15	210	169.355	200 A15	370	1822.66
100 C16	253	204.032	200 A16	370	1822.66
100 C17	266	214.516	200 A17	306	1507.389
100 C18	312	251.612	200 A18	325	1600.985
100 C19	298	240.322	200 A19	298	1467.98
100 C20	240	193.548	200 A20	384	1891.626
Average	276	223	Average	307	1512
Std. Deviation	71	57.5	Std. Deviation	39	191

Table 1Microcrack density results

Residual stress pattern

Figure 8 shows the residual stresses profile for both conventional and accelerated hard chromium. The testing was made by x-ray diffraction and Cr- k_{α} radiation, using the Raystress equipment.¹² Electrolytic polishing was used for surface exposure and layer removal.

As seen in Fig. 8, the accelerated hard chromium exhibits higher tensile residual stresses compared to conventional chromium, particularly at the highest thickness studied. It is well known that for each bath composition the tensile residual stress increases with hard chromium thickness.^{6,11} Pina, *et al.*¹¹ showed that, despite the fact that microcracks result in residual stress relief in the coating, the residual stresses still remain high at the surface (~800 MPa), decreasing as one descends into the core of the deposit (~300 MPa) and increasing again at the interface. At the interface, the values are dependent on the substrate material.

In this study, we observed the same tendency, *i.e.*, the residual

stresses were high at the surface (~700 MPa for accelerated and ~480 MPa for conventional), decreased at half the coating thickness (~500 MPa for accelerated and ~280 MPa for conventional), and showing zero stress at the interface, with the AISI 4340 steel substrate. On the other hand, we also observed that the tensile residual stresses contained in the accelerated hard chromium plating induced compressive residual stresses in the substrate. Horsewell¹³ mentions that the chromium layer contraction during the electroplating process generates high equi-biaxial tensile stresses in the coating and compressive stresses in the substrate. However, in the coating with simple hard chromium layer submitted to fatigue process, the "benefit" given to the substrate is counteracted by the microcrack density contained in the coating, as will be discussed later. On the other hand, we also observed that no compressive residual stresses arose in the conventional hard chromium plated AISI 4340 steel. Thus, from Fig. 8, we can conclude that the higher the microcrack density, the higher the tensile residual internal stress, and vice-versa.

Effect on the fatigue strength

The S-N curves for the rotating bending fatigue tests for both the accelerated and conventional hard chromium electroplated specimens, as well as for uncoated specimens are shown in Fig. 9. First, it is seen that the coating effect is to decrease the fatigue strength of AISI 4340 steel. Yet, the performance of conventional hard chromium is better than that for accelerated hard chromium. This may be attributed to the lower microcrack density of the former. So, from Figs. 8 and 9, the higher the microcrack density, the higher the tensile residual internal stress and the worse the fatigue performance. This means that the accelerated hard chromium electroplating is responsible for higher tensile residual internal stresses and also exhibits the highest crack initiation (stress concentration) and propagation front amount, despite the "benefit" attributed to the substrate related to compressive residual stresses mentioned by Horsewell.¹³ Such a benefit is counteracted by the microcrack density. When submitted to the tensile residual stress action, in addition to external loads, the microcracks propagate and reduce the fatigue life of a component.

In addition, A. R. Jones¹⁴ mentions that deposits of chromium with a low microcrack density have them propagated deeper into the coating. In fact, measurements of the surface roughness of both accelerated and conventional hard chromium indicated values of $R_a = 0.74 \mu m$ and 1.60 μm , respectively. In addition, the excellent adhesion of the hard chromium to the substrate made it easier for crack propagation into the basis material.

Effects on abrasion wear resistance

The abrasion wear resistance of both hard chromium deposits was evaluated according to FED-STD-141C,¹⁵ and the results, in terms of wear weight loss, are represented in Fig. 10. Comparing the results, one sees better performance for the conventional hard chromium up to 9,000 cycles. However, with the parabolic behavior seen with the accelerated hard chromium, after 10,000 cycles, the positions reverse and there is lower wear weight loss than with conventional hard chromium plating. The higher microcrack density in hard chromium, which results in more edges, as well as lower toughness and higher brittleness, may explain the initial increase in the wear weight loss. The higher the crack density, the higher the amount of previously detached solid particles, which are embedded in the microcracks and decrease the wear strength.⁵ This may results in micro-cutting, which is considered to be the predominant wear weight loss mechanism.¹⁶

Effects on salt spray resistance

Qualitative results of the corrosion testing were obtained by visual inspection and image analysis of the specimen surface after exposure to salt spray.¹⁷ Figure 11 clearly shows the higher salt spray resistance of the accelerated hard chromium during all tests.

The accelerated hard chromium plated specimen with 49 μ m thickness showed about 5% corrosion products after 48 hr salt spray exposure. On the other hand, the identical conventional hard chromium electroplated specimen was completely corroded (100%). This behavior is related to the number of microcracks in the deposit, such that the higher the microcrack density in the deposit, the smaller the corrosion penetration. That is, the higher microcrack density, the more uniform the distribution of the corrosion process over the specimen surface.^{3,5} On the other hand, a lower microcrack density implies a more localized concentration of the corrosion process and, consequently, there is a higher local corrosion rate that can penetrate to the substrate.



Figure 8-Residual stress pattern.



Figure 9–S-N curves for rotating bending fatigue tests.^{5,7}



Figure 10–Abrasive wear test results for accelerated and conventional hard chromium electroplating.⁷

In general, the corrosion resistance is also related to the surface roughness of a part, *i.e.*, the higher the surface roughness, the higher the corrosion attack due to higher surface area.¹⁴ Despite the higher microcrack density of the accelerated hard chromium, the surface roughness is lower than that of the conventional hard chromium, as mentioned before. Therefore, the conventional hard chromium electroplating process yields a lower microcrack density and consequently deeper microcracks. It is also clear increased thickness enhanced the salt spray resistance of hard chromium.

Conclusion

In this study a simple image analysis procedure was presented to determine the microcrack density in hard chromium electroplating based on the surface morphology of the coating. We verified the direct correlation between microcrack density and the corrosion, wear and fatigue performance of two different hard chromium deposits, as well as between the residual stress profiles generated. The comparative analysis of two types of hard chromium plating indicates the higher microcrack density, the lower fatigue strength, the better corrosion and wear resistance (up to 10.000 cycles), and the higher the tensile residual stress values. For a 7-to-1 microcrack density ratio between both types of hard chromium plating, the tensile residual stress ratio was about 1.5-to-1. This confirms that the microcracking is an intense residual stress relief process, but which still remains high in the coating. Consequently, this can also limit the maximum coating thickness that can be obtained. Thus, it was concluded that it is possible to estimate and compare the mechanical and chemical properties of hard chromium electroplating, generated from different bath compositions, from its surface morphology. This is particularly important when implementing this simple, rapid procedure during process development. Based on the "in loco" microcrack density, the procedure can be used to control the ultimate chemical and mechanical properties of hard chromium electroplating for a specific industrial application. The image analysis procedure can also be applied on any image obtained by scanning electron microscopy (SEM). Since the same microcrack density tendency is expected, this procedure can be applied to determine the microcrack density as a function of the thickness as well.

Acknowledgment

The authors are grateful to CAPES, Embraer-Liebherr/EDE, FAPESP and FUNDUNESP for financial and technical support.

References

- 1. D.C. Bolles, Welding Journal, 74 (10), 31 (1995).
- T. Khaled, A Look at Hard Chrome Replacement Metallurgy Report #ANM-112N-01-02 U.S. Department of Transportation, Federal Aviation Administration, Washington, DC, 2001.
- 3. M.P. Nascimento, *et al.*, *Plating & Surface Finishing*, **88** (4), 84 (2001).
- M.P. Nascimento, et al., International Journal of Fatigue, 23 (7), 607 (2001).
- 5. M.P. Nascimento, *et al.*, *Surface & Coatings Technology*, **138** (2-3), 113 (2001).
- 6. A.R. Jones, Plating & Surface Finishing, 76 (4), 62 (1989).
- 7. J.M. Tyler, *Metal Finishing*, **93** (10), 11 (1995).
- G. Dubpernell, in *Modern Electroplating*, F.A. Lowenhein, Ed., Wiley Interscience, New York, NY, 1969; p. 80-129.
- 9. F. Durut, et al., Metal Finishing, 96 (3), 52 (1998).



Figure 11–Salt spray test results for hard chromium electroplated samples after 48 $\rm hr.^7$

- K.J. Kurzydlowski & B. Ralph, *The Quantitative Description* of the Microstructure of Materials, CRC Press Inc., Boca Raton, Florida, 1995.
- J. Pina, et al., Surface & Coatings Technology, 96 (2-3), 148 (1997).
- 12. T. Gurova, et al., Scripta Materialia, 36 (9), 1031 (1997).
- A. Horsewell, *Materials Science and Technology*, **14** (6), 549 (1998).
- A.R. Jones, ASM Handbook Corrosion 5th ed. Vol. 13, ASM International, Materials Park, Ohio, 1996: p. 871-875.
- 15. FED-STD-141-86, Abrasion Resistance (Taber Abraser), Method 6192.1,
- 16. H. Wang, et al., Wear, 195 (1-2), 47 (1996).
- ASTM Standard B 117, 1997, Standard Practice for Operating Salt Spray (Fog) Apparatus, ASTM International, West Conshohocken, Pennsylvania.

About the Authors



Dr. Marcelino Pereira do Nascimento is a Researcher in the Materials and Technology Department of São Paulo State University - UNESP/FEG/DMT. His research interests are focused on the effects of surface treatment such as shot peening, electroless nickel and hard chromium coatings on the mechanical properties (fatigue and residual stresses) of high-strength steels used in the

aeronautical industry. He is author of several papers on the subject, with particular emphasis on fatigue and fracture.



Dr. Herman Jacobus Cornelis Voorwald is a Professor in the Materials and Technology Department of São Paulo State University - UNESP/FEG/DMT. His research interests also include the effects of surface treatment such as shot peening, electroless nickel and hard chromium coatings on the mechanical properties (fatigue and residual stresses) of ferrous and non-ferrous materials used in

the aeronautical industry. He has published numerous papers on the subject.